

**OBG Laboratories, Inc.  
QA Program Manual  
Remedial Investigation/  
Feasibility Study**

**National Smelting of New Jersey Site  
Pedricktown, New Jersey**

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Hightstown, New Jersey**

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**O'BRIEN & GERE**

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## I. O'BRIEN AND GERE LABORATORY

### Introduction

For several years the O'Brien and Gere laboratory has been involved in the physico-chemical and microbiological analyses of environmental contaminants for federal, state, municipal and industrial clients. The laboratory has analyzed over 10,000 samples for over 100,000 parameters on an annual basis. The organic and inorganic pollutants occur in several matrices, i.e., potable water, industrial and domestic wastewater, hazardous waste, sludges, sediment, biological tissue, solid, air, etc. The ability to accurately characterize the chemical pollutants in these matrices is paramount.

In this document concepts are presented to outline the laboratory program purpose, policies, organization and operations established to support physico-chemical analyses conducted under USEPA compliance. Implementation of this program will better insure the validity of the data acquisition, and, therefore, will provide a more reliable foundation on which to base decisions. The principles and procedures used are the result of considerations of the general operations and trends in the field of analytical chemistry, analytical instrumentation, statistical quality control techniques, and previous experiences in the laboratory programs conducted under USEPA, local and state government compliance.

## Laboratory Policy

The management of O'Brien & Gere's Laboratory is firmly committed to the Quality Assurance/Quality Control (QA/QC) program depicted in this manual. The program has been implemented and is maintained to assure any data reported by the laboratory are of known and documented quality commensurate with their intended use. The technical personnel who contribute to all or any portion of the laboratory analyses follow the procedures delineated in this manual.

The QA/QC manual is an integral part of a generalized representation of our Good Laboratory Practice program. It is primarily intended to set control guidelines and direction for all the physico-chemical and microbiological measurements performed by the laboratory. The contents of this manual will be re-evaluated yearly by the QA/QC group leader, and if necessary, revisions will be made, and/or the QA/QC program expanded.

A supplementary laboratory manual dealing with specific technical areas has been written and is available to all laboratory personnel. The laboratory manual is reviewed and approved by the QA/QC, Trace Organics and Wet Chemistry group leaders and management prior to distribution to the laboratory staff.

## Quality Control Program Objectives

The primary objective of the O'Brien & Gere Laboratory QA/QC program is to assure the precision and accuracy of all data generated by the laboratory personnel. That is, the data is of known and documented quality.

The QA/QC guidelines are implemented in support of the laboratory surveillance programs and analyses efforts. They reflect the best cost effective effort, and are used to assess, ensure and document that all data collected, stored, reported or used by the laboratory are scientifically valid, defensible and of known precision and accuracy.

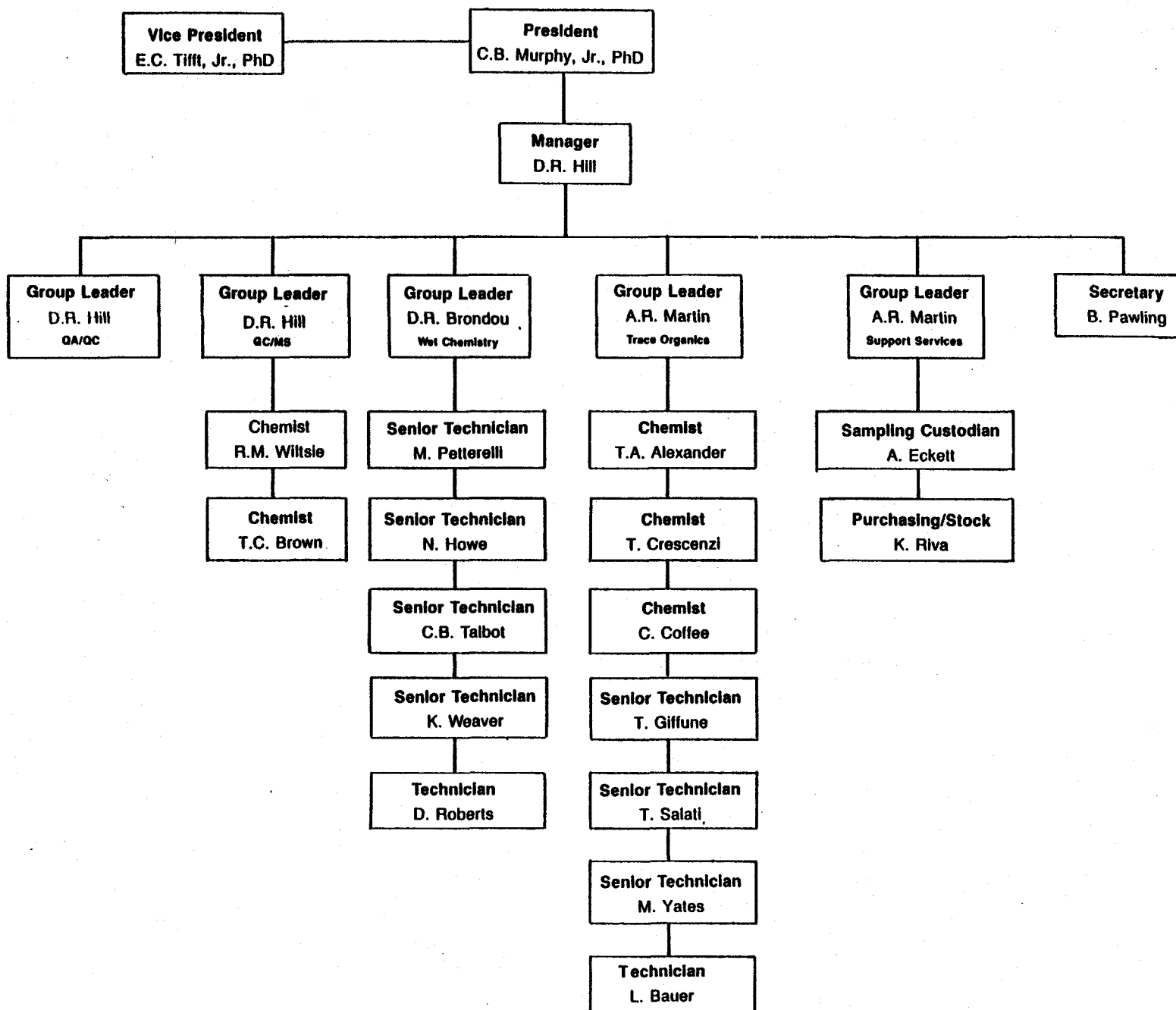
The major effort of the QA/QC program will be to develop a workable day-to-day "QA/QC model", and thus provide the detailed control charts and control limits to measure the laboratory daily performance. The QA/QC activities shall be carried out in accordance with EPA, state and local government mandates. The implementation, coordination and supervision of these procedures will provide the customer with the quality assurance (QA) activities associated with good laboratory practices.

#### Personnel and Organization

Any organization consists of a number of people whose skills and delegated responsibilities assure the quality of the ultimate product, i.e. analytical services. QA/QC procedures commence when the sample is first collected, and continues until the final product is in the client's hand. An organizational chart of the laboratory technical staff is included in Figure 1 to serve as a frame of reference for all QA/QC procedures.

The Laboratory Manager is responsible for the overall administration of the analytical operations at O'Brien & Gere. The section group leaders handle the day to day scheduling and operation, and report to the manager. Together with the group leaders they review

# LABORATORY ORGANIZATION CHART



and approve all policies concerning their specific areas of responsibility.

The QA/QC group leader is responsible for the implementation, monitoring and supervision of the QA/QC program. He assures that the program is conducted in strict adherence to procedures and requirements outlined in this manual. He reports to the Laboratory Manager, and interacts daily with other group leaders and laboratory staff. His duties include:

1. Develops and implements new QA/QC programs, including statistical techniques and procedures.
2. Conducts regular inspections and audits of analytical procedures.
3. Daily monitors accuracy and precision and implements correction measures if "out of control".
4. Maintains copies of all procedures routinely used in the laboratory measurements.
5. Informs management of the status of the QA/QC program by annual status reports.
6. Coordinates and conducts investigations of any customer complaints regarding quality.
7. Reschedule any analysis based on poor accuracy or precision data.

The section group leaders are responsible for the day to day operation and technical questions concerning analytical protocol and together with the QA/QC group leader:

1. Maintain and increase the technical skills of the laboratory technical personnel to achieve optimum quality results.



2. Approve analytical methods, sampling procedures, special QA/QC procedures, and any subsequent revisions in analytical procedures used in their respective areas.
3. Approve completed work.

### Technical Training

All personnel involved in any function affecting data quality (sample collection, analysis, data reduction, and quality assurance) have sufficient technical training (in their appointed positions) to contribute to the reporting of complete and high quality data. The training is achieved through: a) On-the-job training, b) Short-term courses (one week or less), and c) Long-term courses (one semester or longer).

Short and long term courses are available through universities, colleges, and technical schools in statistics, analytical chemistry, and other disciplines. In addition, short-term courses are provided by commercial training organizations, manufacturers of equipment and others.

The trainee and/or analyst performance is evaluated by providing unknown samples for analysis. An unknown, as defined here, is a sample whose concentration is known to the QA/QC group leader or other group leaders but is unknown to the trainee or analyst. Proficiency is judged in terms of accuracy.

## II. GENERAL FACILITIES AND EQUIPMENT

The laboratory is located in the corporate headquarters of O'Brien & Gere in Syracuse. The laboratory maintains a staff of sixteen chemists, biologists and technicians. As many as ten temporary and part-time personnel have been used to meet peak demands. The staff maintains a constant awareness of state-of-the art techniques in environmental analysis through its review of literature. The laboratory has 3700 square feet to utilize for the preparation and analysis of samples and 1200 square feet for receiving and storage of reagents.

The laboratory's involvement in a variety of programs has provided the necessary experience in microbiological, inorganic contaminants and trace organic identification and quantification. Particular expertise has been developed in the area of hazardous waste identification and trace organics analysis including priority pollutants and PCB's. A brief description of available instrumentation, computer services, sample storage and receiving follows.

### Laboratory Instrumentation

The following analytical instrumentation is located in the Syracuse office and has been used on a number of major analytical programs:

(a) Hewlett Packard 5993B Gas Chromatograph/Mass Spectrometer Data System - for the low level identification of organic priority pollutants and other compounds. The unit is equipped with a dual disc, 32K computer and 9-track magnetic tape.

(b) Hewlett Packard 5880A - Gas Chromatograph equipped with dual electron capture detectors. The fully automated system has capabilities for both packed and capillary column work. The system can

operate unattended around the clock to provide rapid turnaround of results.

(c) Tracor Model MT220 gas chromatograph equipped with electron capture and dual flame ionization. The unit is interfaced to a Hewlett Packard Model 3380 S integrator.

(d) Two Tracor Model 550 gas chromatographs, both equipped with Hall electrolytic conductivity detectors, linearized electron capture detectors, and photoionization detectors interfaced to Hewlett Packard Model 3390 integrators.

(e) Due to the highly specialized procedures for cleaning glassware used in the low level analysis of halogenated organics and other substances, a sonic cleaner is utilized. Additionally, a complete glassware supply including Soxhlet extractors, separatory funnels, flasks and chromatographic columns is maintained.

(f) Two Technicon AutoAnalyzers, single and dual channel, for the automated determination of nutrients and other inorganic parameters.

(g) Perkin-Elmer Model 290B Atomic Absorption Spectrophotometer for the determination of metals by flame techniques.

(h) Varian Model 575 Atomic Absorption Spectrophotometer for the low-level detection of metals by conventional flame and graphite furnace (flameless) techniques.

(i) Beckman Model 915 Total Organic Carbon Analyzer, for the determination of organic, inorganic or total carbon.

(j) Dohrman Model DX-20 Total Organic Halide Analyzer, and Model MCTS 20/30 Elemental Analyzer for the determination of chlorine and sulfur in environmental samples.

(k) Bausch & Lomb Model 340 colorimeter, used for those colorimetric procedures not performed on the AutoAnalyzers.

(l) DuPont Model 760 Luminescence Biometer for the determination of adenosine triphosphate (ATP).

(m) Orion Model 4 Specific Ion Meter.

(n) Mettler Model HE10 Electronic Semi-Micro Balance.

(o) Hiack Particle Counter for the determination of particle sizes in water ranging from 0.5m to 300m.

(p) A walk-in refrigerator for storage of samples prior to analysis.

The laboratory also maintains a wide range of the usual supporting equipment such as pH meters, analytical balances, ovens and incubators, refrigerators and hood space.

#### Computer Services

The hardware which serves as the foundation of the firm's computer facilities has been responsible for the ability of the O'Brien & Gere laboratory to store and retrieve all data for individual clients.

The quantity of data has led to the development and utilization of a computer-based data management system. Samples are logged in, analyses are scheduled and output is received, all via time-shared or batch computer programs. One of the benefits of this system is that turnaround time has been reduced to a practical minimum. Data can be reported in a variety of formats. The standard computer output includes sample identification and various test results. A variety of statistical and modeling programs are available for the evaluation and interpretation of data.

### III. GENERAL CONSIDERATIONS

#### Maintenance

A preventative maintenance schedule on all instruments, balances, and equipment requiring maintenance is followed. All maintenance, whether performed by the laboratory or other professional sources, is documented in appropriate log books. Entries are made each time maintenance is performed and include the reason for maintenance, what was performed, by whom, and the dates and initials of the analyst in charge during the maintenance.

#### Calibration

Thermometers needed for critical temperature determination and control are calibrated against an NBS thermometer on site once a year. Analytical balances are professionally calibrated and cleaned once a year and checked with Class S weights daily by analysts who routinely use the balances. Calibration data are entered into a specific calibration notebook, which is kept with the equipment being calibrated. When the balances are professionally calibrated, a document stating the specific balance (model and serial number), its location, and the data calibrated is provided by the company or individual providing such service.

#### Reagent Quality

The quality of reagents and instrument readings are maintained by the following procedures:

(a) Reagents for quantitative purposes are ACS analytical quality grade or better.

(b) Each sample is collected in a new container to minimize contamination. This rule does not apply to bacteriological samples for which sterilized glass bottles are used, or trace organic samples for which solvent rinsed glass bottles are used.

(c) Distilled deionized water with a conductivity not more than 1.5 micromho/cm is used in the preparation of all reagents and for final rinses. The conductivity is measured daily and recorded in the quality control log. The pH is also checked daily and the values recorded.

(d) All volumetric glassware is National Bureau of Standards Class A grade or better.

(e) All glassware is cleaned and rinsed with distilled water and visually inspected before use. Any volumetric glassware found to be etched or cracked is discarded.

(f) The operating temperatures of all ovens, incubators, water baths and refrigerators are recorded daily in the quality control log.

(g) All reagents are discarded after a set interval which has been established and recorded in the Laboratory Handbook.

(h) The date a prepared reagent is made is entered into the Reagent Log and initialed by the preparer. Therefore, the results which have been affected by a contaminated or otherwise improper reagent can be easily determined. These results are either recalculated or discarded and the analysis may be repeated if possible. Reagent containers are also dated when new solutions are prepared and are initialed. These procedures are followed for all (even daily) preparations.

(i) The pH meter is checked with three buffers (4.0, 7.0 and 10.0) and the results are recorded in the quality control log.

## Safety

A safety manual is issued to all laboratory personnel and describes safety policies, procedures and guidelines. Although laboratory workers are trained to be cautious in handling toxic or dangerous materials, they have confidence in the safety features built into their working area, thus enhancing the reliability of their performance.

## Audits and Inspections

The Quality Assurance program is audited weekly for overall adherence to the guidelines and procedures outlined in this manual. The QA/QC group leader is responsible for scheduling and ensuring that each audit occurs.

Monthly meetings are scheduled between the QA/QC group leader and manager of Analytical Services to thoroughly discuss the program. Any corrective action required is monitored and ensured by the QA/QC group leader.

#### IV SAMPLE COLLECTION AND TRACKING

Valid representative samples of environmental matrices are collected through well defined sampling protocols. The sampling may be performed by the laboratory sampling team, or the customer who then assumes responsibility for properly obtaining, handling, preserving and shipping the sample.

##### Sample Collection and Handling

A well defined sampling protocol must ensure that:

- a. sampling team members are competent and qualified
- b. proper sampling methods are used
- c. equipment is accurately calibrated
- d. all samples are properly handled to prevent contamination
- e. samples analyzed are actually the samples collected under reported conditions.

For these reasons, samples are kept in secure places from time of collection until they are analyzed. It is the joint responsibility of the group leader and sampling team leader to ensure that approved methods are used, and it is the responsibility of each sampling technician to assure that the equipment is accurately calibrated.

##### Chain of Custody

The laboratory sampling protocol generally follows a chain of custody procedure. The procedure creates an accurate, written, legally defensible document that can be used to trace possession of sample from its collection through analysis and final disposal.



The basic elements in the chain-of-custody phase of our QA/QC program are:

1. Sample collection and handling
2. Sample analysis
3. Preparation and filing of test report

These measures are documented by the chain of custody form (Figure 2) signed by all handlers of the sample(s). As defined here, a sample is "in custody" if it is:

- a. in actual physical possession, or
- b. in view after being in physical possession, or
- c. in a locked repository, or
- d. in a secure, restricted area.

#### Analysis, Preparation and Filing of Test Report

A critical concern of QA/QC program is the maintenance of sample and data base integrity and the timely preparation of data reports. The data management program allows for the identification of samples and the maintenance of the discrete character of the data generated by each respective sample. This system is a unique advantage over manual methods and has permitted the laboratory to successfully tabulate data involving high numbers of samples and multiple analyses. The system may be divided into the following phases:

1. sample identification -- as each sample enters the laboratory, it is assigned a unique access number found on a sample identification ticket. This identifier permits the discrete organization of all information and data relating to that sample, whether for analytical

## CHAIN OF CUSTODY RECORD

[illegible]

identification purposes, reference in paper-copy records and correspondence, or computer storage and recall.

2. data organization -- in a preliminary planning phase of any analytical investigation involving the laboratory, a computer codification format can be established which can serve as the basis for storage and retrieval of data. This format is characterized by the categorization of samples, with any type of identification permissible for the classification. The categories may be based on any similarities (or dissimilarities) in the total volume of samples.

The storage and retrieval of quality control sample data is also managed with the laboratory's computer-based data management system. Samples are tagged and data is input, stored and retrieved as with any routine project samples. This has been made possible by the use of a unique quality control project number by which such data may be identified.

## V. METHODS AND PROCEDURES

The laboratory analyzes a variety of matrices for a number of different environmental constituents of concern. Therefore, several documents are referenced which include the procedures employed. The following list itemizes the most widely used documents:

1. Standard Methods for the Examination of Water and Wastewater.
2. Methods for Chemical Analysis of Water and Wastewater.
3. ASTM Annual Book of Standards.
4. Code of Federal Regulations.
5. NIOSH Manual of Analytical Methods.
6. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

When analyzing samples by the above standardized methods, the accuracy or precision of the data generated by the laboratory is determined through analysis of replicates, spiked samples, synthetic reference standard samples, and/or field or laboratory blanks along with each set of samples. Any interferences are identified and documented.

In general, the methods accuracy is determining by spiking the sample matrix with the analyte at a minimum of three concentration levels. The range of the spiking levels is selected to bracket the concentration of interest. Percent recoveries of the spikes are calculated and are compared with synthetic standards. The methods precision is determined by analyzing a minimum of three replicates at each spiking level. The precision is evaluated by calculating the standard derivation.

The data generated is, whenever possible, input into the laboratory base data management system. Analyst's work sheets are filed for one year as a temporary record. When approved and signed, data reports and pertinent information are reported to the customer.

## VI. INTRALABORATORY QA/QC PROGRAM

A quality control program is a systematic attempt to assure the precision and accuracy of analyses by detecting and preventing recurrence of errors, or measuring the degree of error inherent in the proven methods used. By identifying the sources of errors confidence in the precision and accuracy of analytical results can be established and improvements in the analytical methods made. To ensure the precision and accuracy of a result our quality control program requires the measurement and analysis of spiked samples, duplicate samples, synthetic standards and blanks.

Duplicate samples are used to provide assurance that the procedure is under control and to determine the statistical limit of uncertainty (i.e., precisions). Synthetic standards and spiked samples are used to determine the quantification of the laboratory accuracy.

In general, our quality control program incorporates the concepts of: a) calibration to attain accuracy, b) replication to establish precision limits, and c) correlation of quantitatively related tests (synthetic standards and spikes) to confirm accuracy.

The overall effectiveness of the program is dependent upon the evaluation of: a) equipment and instruments, b) current state of the art, c) precision of the analytical method itself, d) expected ranges of analytical results, e) control charts to determine trends as well as gross errors, f) data sheets and laboratory procedures adopted for control of sample integrity, g) quality control results on a daily as well as on varying time frames.

## Definitions of Basic Terms

Before we discuss the standard operating practice for the QA/QC program some definitions are in order. These are:

1. Reagent Blank - The reagent (or method) blank is an aliquot of pure, organic free water (or organic reagents) used in the analysis of samples. It is generated by passing the clean matrices through the entire analytical procedure (including all glassware and other materials that come into contact with the sample). These blanks are analyzed along with the samples to verify that: a) qualitatively, no false positives occur, and b) quantitatively, concentrations are accurate and do not reflect contamination.

2. Field Blanks - These are water blanks sent from the laboratory to the sampling site and are returned to be analyzed in the same manner as the samples. If the samples are to be analyzed for purgeable organics, the analysis of field blanks provide a check on possible contamination of the samples by permeation of volatiles through the septum seal. If positive interferences occur the analytical results are rejected unless sufficient data can be obtained from these blanks to allow correction of results.

3. Duplicates - Duplicates are the result of splitting a field sample into equal amounts and are treated throughout as two unique samples. The results of duplicate (or replicate) analyses provide information on the overall precision of the analytical methodology. Quantitative results are obtained by calculating the relative percent difference (RPD) for each analyte in the sample matrix.

4. Spike - Spikes are the result of the addition of a known amount of analyte to a sample or a blank. The analytical results yield

a quantitative measure of accuracy (spiked blanks) or percent recovery (spiked samples). The measured accuracy reflects the best result which can be expected, whereas the percent recovery reflects matrix effects upon the analytical method accuracy.

Because several different environmental matrices are analyzed (e.g., potable water, effluent and influent waters, process wastes, sludges, etc.), two spiking levels are necessary when analyzing different samples. Relatively clean samples are spiked at detection limit and 10 times the detection limit for each component. Highly polluted samples are spiked at 100 times the detection limit for each component. Ideally, the spike should be 50 - 100% of the original concentration of each analyte in the sample matrix. If the added spike is less than 10% of the sample result, the data are questionable and statistically unacceptable.

5. Surrogate Spike - These are the result of the addition of known amounts of standards to every sample prior to the analysis. The standards are chemically similar to the compounds in the fraction being analyzed. In addition, some standards added have compounds which are not likely to be found in environmental samples. The analyses of surrogate spikes provide quality control on every sample by constantly monitoring unusual matrix effects, gross sample processing errors; etc. These spikes are not used as internal standards for quantitation.

6. Reference Standard (reference audits) - These are the analysis of independently prepared standard solutions or synthetic standards. Two types of standards are used, i.e., a) internal reference standard solutions (synthetic standards prepared in-house), and b) external



reference standard solutions obtained from outside sources (i.e., primarily EPA).

The external audits samples are used for monitoring the complete analytical method. These samples are introduced at the onset of the procedure (typically extractions) and carried through the entire analysis.

The internal standard audits are used to verify the "accuracy" of quantitative instrument calibration. All standard solutions are prepared by the QA/QC group leader and are submitted blind for analyses. The analyst analyzes the solutions as discrete samples and a percent recovery or percent error is calculated. Errors greater than 5% are carefully investigated and differences resolved through proper action.

#### Guidelines for Evaluating the QA/QC Program

This section defines the QA/QC program for the analysis of environmental pollutants, i.e., the analysis of trace organics by gas chromatographic (GC) and GC/MS techniques, and analysis of inorganic pollutants by wet techniques and atomic absorption (AA), etc. The QC program for the analysis of trace organics by GC and GC/MS is different due to the unique nature of the analytical problems addressed by the GC/MS methodology. Therefore, the QC requirements for these two techniques will be addressed separately. A description of the QC program follows.

##### 1. Gas Chromatography

In general, when GC methodologies are used the specific analyte or class of analyte is known. As a result a more specific, less generalized QC program can be defined. For example, accuracy data can be

collected prior to analysis of actual samples, and often previous QC data for a particular analyses is available.

The QC program outlined below depicts the procedures used to determine the quality of the data generated in the trace organics analyses. The steps monitored include extractions, concentration, qualitative and quantitative analyses and confirmation.

a) Method Verification

The methods are validated before they are used in routine analysis of samples. Method validation includes analysis of reagent blanks, blanks spiked with compound(s) of interest, analytical standards and standard mixtures. The results from these analysis approximate the best data to be expected from the method.

The extraction and concentration steps are validated by spiking a minimum of 2 blank samples with the same matrix as the sample of interest. The concentration of the analyte used for the spiking is 10 times the detection limit. The accuracy (or percent recovery) of the method is calculated by:

$$\text{ACCURACY} = \frac{(\text{spiked sample result}) \times 100}{\text{spike added}}$$

and is recorded on transcription sheets and is assigned a unique QC number. The data is then logged and stored in the computer.

b) Instrument Calibration and Performance

To insure good analytical data the analytical instruments are calibrated prior to sample analysis by analyzing three standards of analyte which span the suspected concentration range of the analyte in the sample. The performance of the instruments are checked by analyzing a standard mixture. If the retention time or

area counts vary more than 10% from previous calibration the standard mix is reanalyzed. If the deviation is still more than 10%, a new standard mix is analyzed. If the new standard mix still yields greater than 10% deviation, instrument malfunction is suspected and proper action is taken to resolve the problem.

### Routine Analysis

The quality of the analytical data generated during routine analyses is monitored by the following:

- 1) Contamination from reagents and glassware is identified by analyzing a reagent blank. One reagent blank is prepared for every 20 or fewer samples analyzed (or when a new lot of reagent is used in the analysis).

- 2) The analytical method accuracy is determined by spiking a known amount of analyte into a sample and blank. The percent recoveries are then calculated. The amount of analyte recovered from the blank indicates the best result which can be expected from the method. The amount of analyte recovered from a sample reflects matrix effects upon the accuracy of the method. Two spikes are prepared for every 20 or fewer samples analyzed.

- 3) The analytical method precision is determined by analyzing equal amounts of a split sample. Ideally, the analytical results will be identical; however, differences occur due to variations in the procedure. A quantitative measure of these differences is assessed by calculating the relative percent differences (RPD) for each analyte in the matrix and the results compared.

In general, one duplicate is analyzed for every 20 or fewer samples, and the performance of the analytical instrument verified. Whenever possible identification is confirmed by a second procedure.

#### GC and GC/MS Characterization of Trace Organics

The requirements for the characterization of trace organics analyses include: 1) the identification and quantitation of unknown pollutants, 2) the specific detection of selected groups of pollutants (i.e., Priority Pollutants by GC/MS), and 3) other analyses requiring GC/MS for identification, verification and/or quantitation. A summary of the required audits is given in Table I. The performance and calibration of the GC and GC/MS systems are monitored and maintained on a regular basis by the procedures and methods discussed below.

TABLE I. SUMMARY OF SAMPLE ANALYSIS AUDITS REQUIRED FOR THE CHARACTERIZATION AND QUANTITATION OF TRACE ORGANICS

AUDIT	AUDIT
Spike	Mass Spectrometer:
Reagent Blank	mass calibration
Duplicate Sample Analysis	response calibration
Standard Mix	standards
Reference Standard	Computer Match
Standards and Calibration Curve	Reference Spectra Comparison
GC Retention Times	Completeness and Accuracy
GC Peak measurement calculation	

## 1. Calibration of GC/MS System

At the beginning of each day the GC/MS system is calibrated and tuned by examining the mass spectrum of decafluorotriphenylphosphine (DFTPP) or 4-bromofluorobenzene (BFB). The details are discussed below.

### a. Base/Neutrals (and Acids or Pesticide) Fractions

The analysis of 50 nanograms of DFTPP is carried out daily by direct injection into the GC inlet. The resulting mass spectrum is then examined. The requirement is that the mass spectrum of 50 nanograms DFTPP must meet the specification of the key ions and ion abundance criteria listed in Table II.

### b. Volatile (Purgeable) Fraction

The analysis of 20 nanograms of BFB is carried out by direct injection into the GC/MS. The requirement is that the mass spectrum of 20 nanograms BFB must meet the prescribed specifications of the key ions and ion abundance criteria listed in Table II.

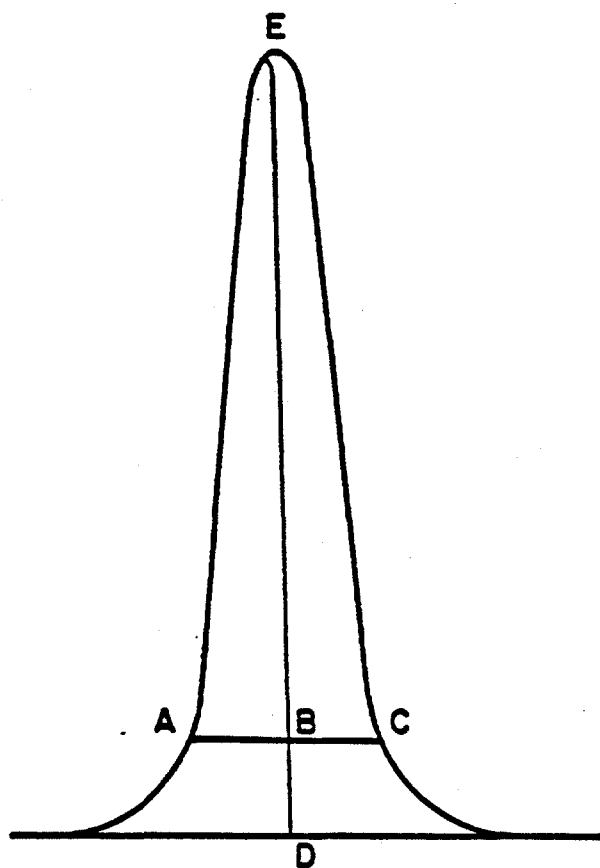
## 2. GC Column Performance Check

The GC columns performance are checked at the beginning of each day that samples are analyzed. For base/neutrals and acid fractions the columns performance are monitored by injecting 100 nanograms (ng) of benzidine and pentachlorophenol, respectively. For purgeables the column is checked by injecting 20 ng of BFB. Performance acceptance is based on calculations of tailing factors (see Table III).

TABLE II. KEY IONS AND ION ABUNDANCE CRITERIA FOR DFTPP AND BFB

DFTPP		BFB	
MASS	ION ABUNDANCE CRITERIA	MASS	ION ABUNDANCE CRITERIA
51	30-60% of mass 198	50	20-40% of mass 95
68	less than 2% of mass 69	75	50-70% of mass 95
70	less than 2% of mass 69	95	base peak, 100% relative abundance
127	40-60% of mass 198	96	5-9% of mass 95
197	less than 1% of mass 198	173	less than 1% of mass 95
198	base peak, 100% relative abundance	174	70-90% of mass 95
199	5-9% of mass 198	175	5-9% of mass 95
275	10-30% of mass 198	176	70-90% of mass 95
365	greater than 1% of mass 198	177	5-9% of mass 95
441	less than mass 443		
442	greater than 40% of mass 198		
443	17-23% of mass 442		

TABLE III EXAMPLE CALCULATION OF TAILING FACTOR



$$\text{TAILING FACTOR} = (BC/AB)$$

Example calculation:

Peak Height = DE = 100 mm

10% Peak Height = BD = 10 mm

Peak Width at 10% Peak Height = AC = 23 mm

AB = 11 mm

BC = 12 mm

Therefore: Tailing Factor =  $(12/11) = 1.1$

## Wet Chemistry and Bacteriology

The quality of the analytical data generated from inorganic and microbiological analyses of environmental contaminants are monitored as follows:

### 1. Wet Chemical Instrumental Methods

The atomic absorption (AA) spectrophotometer and AutoAnalyzer are calibrated using appropriate calibrating standards and blanks. The calibrations are checked by analyzing synthetic standards at five different concentration levels. The results are used to generate standard curves by least squares fit of the data via computer programs. The deviation of the standards from the least squares fit (standard curves) and the standard deviation of the fit are printed on the daily printout and the data stored accordingly in appropriate computer data bases. If deviation from accepted values occur analyses of sample and instrumental calibrations are repeated. Standard curves are generated regularly.

For colorimetric analyses that do not use the standard curve program, one or more standards are analyzed with each group of samples. The results are compared to generally accepted criteria, i.e., percent recovery (or percent error) and relative percent error.

Spectrophotometric instruments are checked by comparing the gain settings or percent transmittance for known (synthetic) standards to previous values. This monitoring method shows any decrease in sensitivity or other systematic effects in performance.



The conductivity meter is checked each time a group of samples is analyzed. The conductance of a standard solution is entered in the quality control log. In addition, the cell constant is checked annually by measuring the electrical conductivity of potassium chloride reference solution. The results are also entered in the quality control log book.

## 2. Bacteriology Techniques

Quality control extends to all aspects of the bacteriological laboratory. The date of preparation of media and the various solutions used in analysis are recorded in the quality control log together with any information which may be important to its preparation such as pH, lot or control number, manufacturer and concentration. In addition, random samples of prepared media are incubated under the same conditions as unknown samples to insure the maintenance of sterility during preparation and use.

The efficiency of autoclave sterility is monitored by the monthly use of Kilit ampules (BBL), a suspension of Bacillus stearothermophilus spores. The sterility of rinse water is checked periodically by the filtration and incubation of a reagent blank (sterile rinse water).

As part of the overall quality control program, the bacteriological quality of the distilled deionized water supply of the laboratory is monitored weekly. Samples for the standard plate count are taken from the water system prior to entry to the deionization cartridge (following

distillation), after deionization and from the storage tank. The results are recorded in the quality control log. Additionally, the Suitability Test as described in Standard Methods is performed on a yearly basis by an outside laboratory qualified to undertake this testing. Bacteriological samples are included in the duplicate analyses program described in the chemical section.

Humidity checks are performed monthly on Standard Plate Count petri dishes to determine percent moisture loss upon incubation.

## VII. INTERLABORATORY QUALITY CONTROL

To indicate how well our laboratory is performing by comparison with other laboratories performing similar work, O'Brien & Gere Laboratory participates in a variety of proficiency and roundrobin tests. Successful performance in the proficiency analyses of samples results in the laboratory certification.

### Certification

The U.S. Environmental Protection Agency certifies state laboratories to conduct their own intrastate program of certification for the proficiency of private laboratories in potable water analysis. The EPA only certifies private laboratories directly in those states which have not assumed primacy. In New York State, the certifying agency is the NYS Department of Health. The firm's laboratory was one of the first participants in the New York State program and has been certified for chemical, atomic absorption, bacteriological and gas chromatographic analysis of potable water since 1974. Laboratory certification has been extended to the State of Massachusetts and interim states in the State of New Jersey for potable water and wastewater testing requirements.

In addition, the laboratory participates in the round robin analyses of reference samples supplied by the EPA and in the analysis of commercially available reference samples.

## VIII. DEFINITIONS OF STATISTICAL TERMS

The following statistical term definitions are used to identify statistical reports and evaluations:

a. Accuracy and Precision - Accuracy is a measure of the nearness of an analytical result, or a set of results, to the true value. It is usually expressed in terms of error, bias, or percent recovery (PR).

Normally the term "accuracy" is used synonymously with "percent recovery". It describes either the recovery of a synthetic standard of known value, or the recovery of known amount of analyte (spike) added to a sample of known value. The percent recovery (PR) or "accuracy" can be calculated by using:

1. standards:  $PR = (\text{observed value} / \text{true value}) \times 100$

2. spikes:  $PR = \frac{(\text{conc. spike} + \text{sample}) - \text{sample}}{\text{conc. spike}} \times 100$

Precision refers to the agreement or reproducibility of a set of replicate results among themselves without assumption of any prior information as to the true result. It is usually expressed in terms of the deviation, variance, or range. Good precision often is an indication of good accuracy, however, one can obtain good precision with poor accuracy if systematic (determinate) errors are present in the method or instrument used. Systematic errors are either positive or negative in sign. Other analytical errors are indeterminate (random) errors. These are inherent in the analytical methods due to uncertainties in measurements.

b. Average - The average or arithmetic mean ( $\bar{X}$ ) of a set of n values- ( $X_i$ ) is calculated by summing the individual values and dividing by n:

$$\bar{X} = \left[ \sum_{i=1}^n X_i \right] / n$$

c. Range - The range ( $R_i$ ) is the difference between the highest and lowest value in a group. For n sets of duplicate values ( $X_2, X_1$ ) the range ( $R_i$ ) of the duplicates and the average range ( $\bar{R}$ ) of the n sets are calculated by:

$$R_i = |X_2 - X_1|$$

and

$$\bar{R} = \left[ \sum_{i=1}^n R_i \right] / n$$

d. Standard Deviation and Variation - The standard deviation (S) of a sample of n results is the most widely used measure to described the dispersion of a data set. It is calculated by using the equation

$$S = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}$$

where  $\bar{X}$  is the average of the n results and  $X_i$  is the value of result i. Normally,  $\bar{X} \pm S$  will include 68% and  $\bar{X} \pm 2S$  about 95% of the data in a normal distribution curve.

The variance is equal to  $S^2$ . The relative standard deviation (RSD) or coefficient of variation (CV) is the standard deviation divided by the mean and multiplied by 100, i.e.,

$$CV = 100S/\bar{X}$$

It is interesting to note that the precision is increased (value of S reduced) by increasing the number of duplicate analysis. The greater the number of replicate analysis, the greater the statistical confidence that the true mean lies within certain limits about the experimental mean.

e. Standard Calibration Curves - standard calibration curves are widely used in the analysis of inorganic pollutants. These curves are generated from the results of analyses of three or more standard solutions of known concentration and a blank. Typically, they are plots of the instrument response versus concentration. A plot is defined as linear, i.e., obeys the linear equation  $Y=a + bX$ , if the correlation coefficient (R) calculated from the linear regression analysis is 0.996 or greater.

The intercept (a), slope (b) and correlation coefficients ( $R_c$ ) can be calculated from:

$$a = \frac{\Sigma X^2 \Sigma Y^2 - \Sigma X \Sigma Y}{n \Sigma X^2 - (\Sigma X)^2}$$

$$b = \frac{n \Sigma XY - \Sigma X \Sigma Y}{n \Sigma X^2 - (\Sigma X)^2}$$

$$R_c = \frac{\sum (X_i - \bar{X})^2 (Y_i - \bar{Y})^2}{\sqrt{\sum (X_i - \bar{X})^2 \sum (Y_i - \bar{Y})^2}}$$

We fit the analytical data to a linear regression analysis by using a computer program.

f. Absolute and Relative Errors - An absolute error is the difference between the experimental result and the true value. The relative error is the absolute error divided by the true value and multiplied by 100 to yield the percent relative error (PRE). When the true value is not known, the PRE is a measure of the difference (range) of a replicate analysis divided by the mean of the replicate value and multiplying by 100. That is, for duplicates

$$PRE = \frac{100 |X_2 - X_1|}{(X_2 + X_1)/2} = \frac{100 |X_2 - X_1|}{\bar{X}_j}$$

g. Skewness and Kurtosis - Skewness and kurtosis are the numbers used to understand the shape of a given curve. Our groups are data bases of spikes, duplicates, and knowns. The data points in these groups should fall within a normal curve. Aberrations from the normal curve are detected in values of skewness and kurtosis.

Skewness defines the symmetry of a curve. A symmetrical curve must have a skewness of zero. Positive or negative values denote lack of symmetry. Kurtosis defines the peakedness of a curve. A normal distribution curve will have a kurtotic value of 3. Peaked curves will have values greater than three, and broad flat curves will have values

less than 3. These values are monitored by the QA/QC group leader. When aberrant values are noted, the interpretation is usually related to very high or low QC values entering data bases or the persistence of patterns of consistently high or low QC values. It is the QA/QC coordinator's responsibility to research the causes of excessive values and patterns and, where possible, rectify the analytical conditions leading to them.

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#### References

- 1) "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," March, 1979 (EPA-600/4-79-019)
- 2) "Manual of Analytical Quality Control for Pesticides and Related Compounds in Human and Environmental Samples," January, 1979 (EPA-600/1-79-008)



## IX. STATISTICAL QUALITY CONTROL AND THE "DAILY QC MODEL"

Random (indeterminate) and systematic (determinate) errors are inherent in all analytical methods due to uncertainties in measurements. The measurement of physico-chemical and microbiological properties of pollutants in various environmental matrices involve uncertainties which cannot be entirely eliminated. The errors in these measurements, however, can be reduced to tolerable limits by examining and controlling the significant variables.

Additional errors, often unrecognized, are introduced by interfering chemical reactions and other undesirable physico-chemical effects. In many instances absolute values cannot be attained directly.

Although uncertainties cannot be reduced to zero, they can be minimized by using available statistical methods. Estimates of the accuracy (probable "true value") and precision (range of measurement error) can be made for the various analytical methodologies by analyzing blanks, duplicates, spikes and synthetic standards. After sufficient QC data are collected various statistical methods are used to evaluate the quality of data by calculating control and warning limits. A discussion of the statistical methods used follows.

### Control Charts

Control charts provide the necessary tool for detecting quality variations in the various analytical methodologies used for the quantitation of environmental pollutants. They are a continuous graphic indication of the state of an analytical procedure with respect to quality, and assist in deciding when and how to take corrective action. The QC charts are generated for each pollutant from the statistical

evaluation of QC data. A minimum of 15 duplicates and spiked samples and/or synthetic standard analyses are required to generate a control chart.

The control limits (CL) on QC charts are paramount criteria for assessing the significance of variations in the analytical results. For instance, when the plotted QC indicators (i.e., percent recoveries, relative percent error, etc.) fall within these limits, the analytical methodologies used are under "control". If, however, a QC indicator value falls outside the CL's, there is an indication that some assignable cause is present which has thrown the system "out of control". Thus, control limits can be considered warning or action limits. They enable us to detect deviations in analytical procedures, and therefore, take corrective action before producing erroneous results (or results which exceed the absolute maximum tolerable limits).

Common practice set warning limits (WL) at  $\pm 2$  standard (S) deviations (95% confidence level of the normal distribution curve) and control limits (CL) at  $\pm 3S$  limits (99.7% confidence level of the normal distribution curve) on each side of the mean. The CL and WL are calculated from the QC data of duplicates analyses by using the equations and statistical factors listed in Table IV. These CL's and WL's include approximately the entire data set under "in control" conditions, and are equivalent to the commonly used  $\pm 3S$  and  $\pm 2S$  limits, respectively. The qualitative relationship between upper and lower control limits, upper and lower warning limits, and the mean is shown in Figure 3.

TABLE IV STATISTICAL FACTORS AND EQUATIONS FOR CALCULATING QC  
(X BAR AND R) CHART LINES<sup>1</sup>

Observations in Subgroup (n)	Factor			
	A <sub>2</sub>	d <sub>2</sub>	D <sub>3</sub>	D <sub>4</sub>
2	1.88	1.13	0	3.27
3	1.02	1.69	0	2.58
4	0.73	2.06	0	2.28
5	0.58	2.33	0	2.12
6	0.48	2.53	0	2.00
7	0.42	2.70	0.08	1.92
8	0.37	2.85	0.14	1.86

Upper control limit for  $\bar{X} = UCL_{\bar{X}} = \langle \bar{X} \rangle + A_2 \bar{R}$

Lower control limit for  $\bar{X} = LCL_{\bar{X}} = \langle \bar{X} \rangle - A_2 \bar{R}$

Upper warning limit for  $\bar{X} = UWL_{\bar{X}} = \langle \bar{X} \rangle + (2/3) A_2 \bar{R}$

Lower warning limit for  $\bar{X} = LWL_{\bar{X}} = \langle \bar{X} \rangle - (2/3) A_2 \bar{R}$

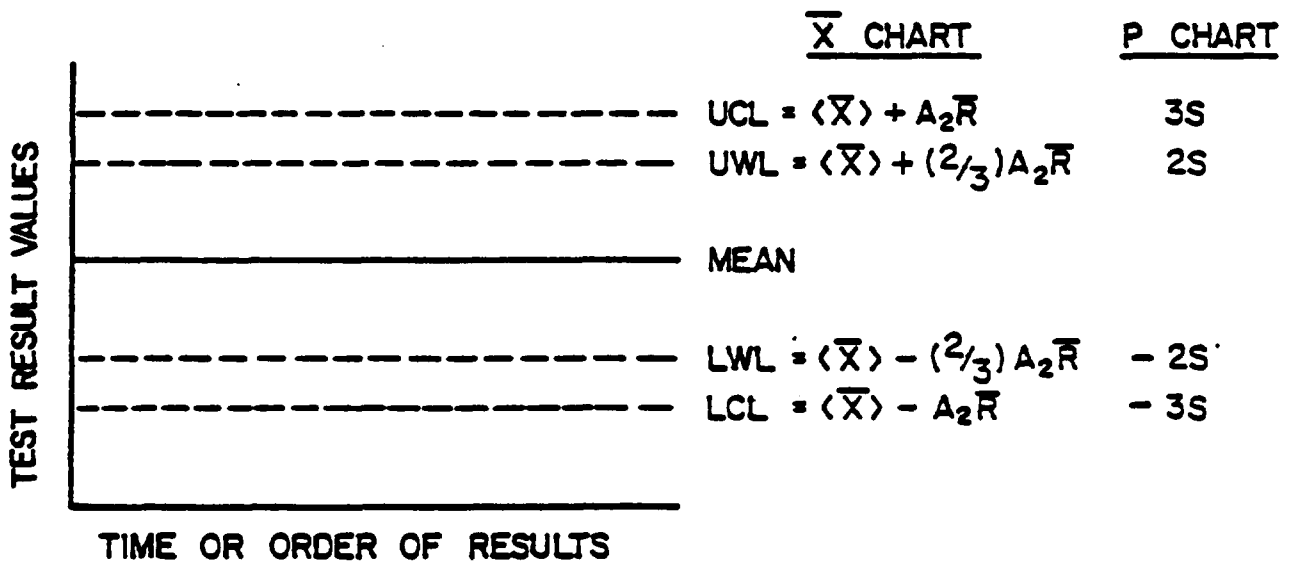
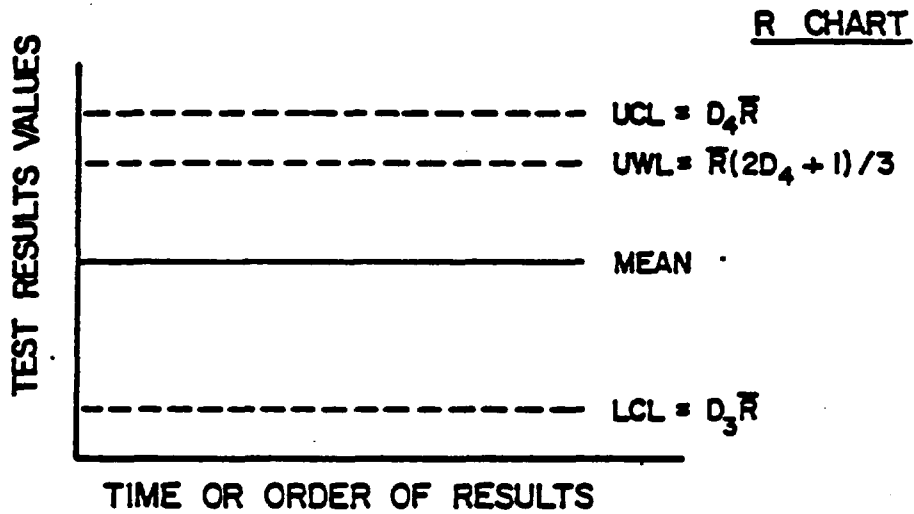
Upper control limit for R =  $UCL_R = D_4 \bar{R}$

Lower control limit for R =  $LCL_R = D_3 \bar{R}$

Upper Warning Limit for R =  $UWL_R = \bar{R} + (2/3)(D_4 \bar{R} - \bar{R})$   
 $= \bar{R} (2 D_4 + 1)/3$

<sup>1</sup> Taken from (1) "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", March, 1979 (EPA-600/4-79-019); and (2) C. Samson, P. Hart and C. Rubin, "Fundamentals of Statistical Quality Control", Addison-Wesley (Massachusetts, 1970), p. 40.

FIGURE 3  
ESSENTIALS OF CONTROL CHARTS



## Statistical Calculations

The statistical techniques used in generating the data for  $\bar{X}$  and R QC charts involves complex mathematics. The short cut methods for calculating the  $\bar{X}$  and R limits are based on the equations listed in Table IV. The statistical factors  $A_2$ ,  $D_3$ ,  $D_4$ , etc. have been calculated by statisticians such that the CL limits involve a maximum risk of making an error only 0.1% to 0.3%. Thus, when the QC charts indicate that the analytical system is "out of control" 997 times out of 1,000 it is likely that something has actually gone wrong and corrective actions are needed. The factors are calculated to yield 3S limits. Examples of QC data and the statistical techniques used to calculate precision and accuracy QC charts follow.

### Precision QC Charts ( $\bar{X}$ and R Charts)

These charts are developed by using a minimum of 15 to 25 QC data results on duplicate analyses. Once these data have been collected over an extended period of time the warning and controlling limits on the QC charts are calculated by using the equations and statistical coefficients listed in Table IV. The procedure used follows:

(1) For each duplicate sample analysis calculate the range ( $R_j = |X_2 - X_1|$ ) and the average ( $\bar{X}_j = (X_2 + X_1)/2$ ) of the concentration of the duplicate set.

(2) Calculate the relative percent range ( $R_j^1$ ) defined as

$$R_j^1 = \text{PRE}/100 = R_j/\bar{X}_j$$

where PRE is the relative error defined in Section VIII.

(3) Calculate the mean ( $\bar{R}^1$ ) relative range by summing the  $R_j^1$  values and divide by the total number (n) of duplicate sets, e.g.,

$$\bar{R}^1 = \left[ \sum_{j=1}^n R_j^1 \right] / n$$

(4) Calculate the grand average  $\langle \bar{X} \rangle$ , i.e., the average of the average of n sets of duplicate averages  $\bar{X}_j$  by using:

$$\langle \bar{X} \rangle = \left[ \sum_{j=1}^n \bar{X}_j \right] / n$$

(5) Calculate the warning and control limits for R and  $\bar{X}$  (see Table IV) by using:

For R:

$$\begin{aligned} \text{UCL} &= D_4 \bar{R}^1 = 3.27 \bar{R}^1 \\ \text{LCL} &= D_3 \bar{R}^1 = 0 \\ \text{UWL} &= \bar{R}^1 (2D_4 + 1)/3 = 2.51 \bar{R}^1 \end{aligned}$$

For  $\bar{X}$ :

$$\begin{aligned} \text{UCL} &= \langle \bar{X} \rangle + A_2 \bar{R} = \langle \bar{X} \rangle + 1.88 \bar{R} \\ \text{LCL} &= \langle \bar{X} \rangle - A_2 \bar{R} = \langle \bar{X} \rangle - 1.88 \bar{R} \\ \text{UWL} &= \langle \bar{X} \rangle + (2/3) A_2 \bar{R} = \langle \bar{X} \rangle + 1.25 \bar{R} \\ \text{LWL} &= \langle \bar{X} \rangle - (2/3) A_2 \bar{R} = \langle \bar{X} \rangle - 1.25 \bar{R} \end{aligned}$$

where for duplicates  $D_3 = 0$ ,  $D_4 = 3.27$ , and  $A_2 = 1.88$  (Table IV); UCL and LCL are the upper and lower control limits, respectively; and UWL and LWL are the upper and lower warning limits. The WL's and CL's correspond, respectively, to the 95% (2S) and 99.7% (3S) confidence limits of a normal distribution curve.

(6) Graph the  $\bar{R}^1$ , UCL, LCL and UWL on the QC charts with appropriate scales which allow additions of new results (Figure 3) and the individual ( $R^1_j$ ) QC data results.

(7) Graph the  $\langle \bar{X} \rangle$ , UCL, LCL, UWL, and LWL on the QC charts with appropriate scales which allow additions of new results and individual ( $\bar{X}_j$ ) QC data.

(8) If QC values are "out of control", i.e., lie outside the control limits, take appropriate corrective action.

#### Accuracy QC Charts (P Charts)

The P charts are the same as the  $\bar{X}$  and R charts since their function is to enable us to detect changes in the laboratory daily performance of analyses and take corrective action. The P-QC charts utilize the sigma (i.e., standard deviation, S) as a quantitative measure of the degree of variations in the analytical methodologies.

The accuracy of the laboratory analytical methodologies is monitored via the analysis of various spiked samples and/or audits of synthetic standards. Spiked samples are also analyzed vis a vis field samples and the percent recovery calculated. Once a minimum of 15 QC recovery data have been collected over a period of time the warning and controlling limits are calculated and P charts developed. The procedure used follows:

(1) For each spiked sample analyzed calculate the percent recovery (PR) using the equations given in Section VIII.

(2) Calculate the mean percent recovery ( $\bar{PR}$ ) by summing the total number of PR's and divide by n (see Section VIII).

(3) Calculate the standard deviation (S) from the percent recoveries -(see Section VIII).

(4) Calculate the warning (WL) and control (CL) limits by using:

$$CL = \text{mean} \pm 3S$$

$$WL = \text{mean} \pm 2S$$

where CL and WL denote, respectively, the upper and lower control limits, and the upper and lower warning limits; S the standard deviation; and mean the average percent recovery ( $\bar{P}\bar{R}$ ) for n spiked samples or synthetic standards. The WL and CL on the accuracy charts (similar to the precision charts) correspond, respectively, to the 95% and 99.7% confidence limits of a normal distribution curve.

(5) Graph the mean, WL, CL and the individual (PR) QC data results on the accuracy chart using appropriate scales.

(6) If QC values lie outside the control limits, the analytical method is "out of control" and appropriate corrective actions are taken.

### The "Daily QC Model"

The "Daily QC Model" comprises two unique activities of our QA/QC program, i.e., the data management and monitoring specific statistical programs of data management systems on a daily basis. The salient features of the programs are discussed below.

#### 1. Data Management

Integral to the laboratory's QA/QC program is the management of data generated from specified quality control procedures. These procedures are designed to monitor all laboratory analyses and ultimately, to ensure the highest possible quality of results. As



previously mentioned, the duplicate, the spiked recovery, the synthetic known and the blank(s) are the analytical tools used to monitor the precision and accuracy of analytical methods. Recall:

- (a) duplicate analyses monitor analytical method precision,
- (b) spiked samples and synthetic knowns monitor analytical accuracy, and
- (c) analyses of blanks account for possible sources of contamination.

The data produced from these tests is maintained via a quality control data management system which has the dual function of relating QA/QC data to analytical performance on a daily as well as varying time frames.

The key to the management of QA/QC data in the laboratory is the Firm's Honeywell X560 computer. Quality control computer programs allow for the calculations, storage, segregation, interpretation, monitoring and retrieval of each bit of QA/QC information. A discrete system of sample identification is used which allows the computer to perform these functions automatically. Each QA/QC sample is assigned a specific code identifying it as a blank, duplicate, spike or synthetic known sample. The code identifiers place each QC value in an appropriate data base which provides a permanent record of each and every quality control sample. These data base are then used as the starting point of various statistical analyses of QC data which aid in understanding the developed analytical information.

Specific statistical programs are available for the various types of QA/QC samples, and generate precision ( $\bar{X}$  and R) and accuracy ( $\bar{P}$ ) quality control charts. These charts provide the graphic

representation of the QA/QC information and are used to monitor the accuracy and precision of the various analytical methodologies daily.

## 2. Monitoring Statistical Programs of Data Management Systems

The QA/QC programs are made available to the QA/QC group leader and the analyst to allow daily response to analysis. The programs offer instant presentation of statistical values which are checked vis a vis the most recent mean, standard deviations and control limits calculated from each data base in the computer. As a result the QA/QC group leader and the analyst will know immediately whether or not the analytical method performance is in control (lie within acceptable ranges) and a decision can be made to accept, reject or repeat the analysis.

In addition, a program exists for the QA/QC group leader which presents all quality control information in a daily printout (see Figures 4 and 5). On this printout, information concerning QC samples is organized for review by the QA/QC group leader. The sample number, the test parameter, the QC sample type, the date of analysis, percent recoveries, relative errors and all values necessary for the calculation of QC data are collected on this printout (Figure 4). In addition to the QC values, commensurate warning and control limits are given. The QA/QC group leader is able to examine these data for acceptability. A quick scan can tell him the status of unfinished samples and values of QC data entering data bases. It is at this point where errors are detected, researched, and corrected whenever possible. We feel that the use of this monitoring program minimizes elapsed time between analysis and data review, therefore, greatly

TABLE V. SUMMARY OF VARIOUS QA/QC ITEMS  
ON DAILY COMPUTER PRINTOUT

ITEM	INFORMATION
CONTROL CHARTS	X Bar and R Charts (precision) P Charts (accuracy)
TABLES	Blanks Duplicates (Percent Relative Error) Spikes (Percent Recovery) Synthetic Standards (Percent Error)
WARNING PROGRAM	Outliers on all QC Data Base Mean and Standard Deviation Upper and Lower Warning and Control Limits
STATISTICS	Average, Mean and Standard Deviation Upper and Lower Warning and Control Limits Skewness and Kurtosis Percent Relative Error Percent Recovery Percent Error

improves the sensitivity of our QC program to our analyses. The earlier errors are detected and corrected, the less time is required to deliver valid results to a client.

A summary of the various QC activities and statistical calculations found in the daily printout is given in Table V. If QC values are found to lie outside the control limits, corrective actions are taken to bring the analytical method "under control". The various corrective actions are delineated in Table VI.

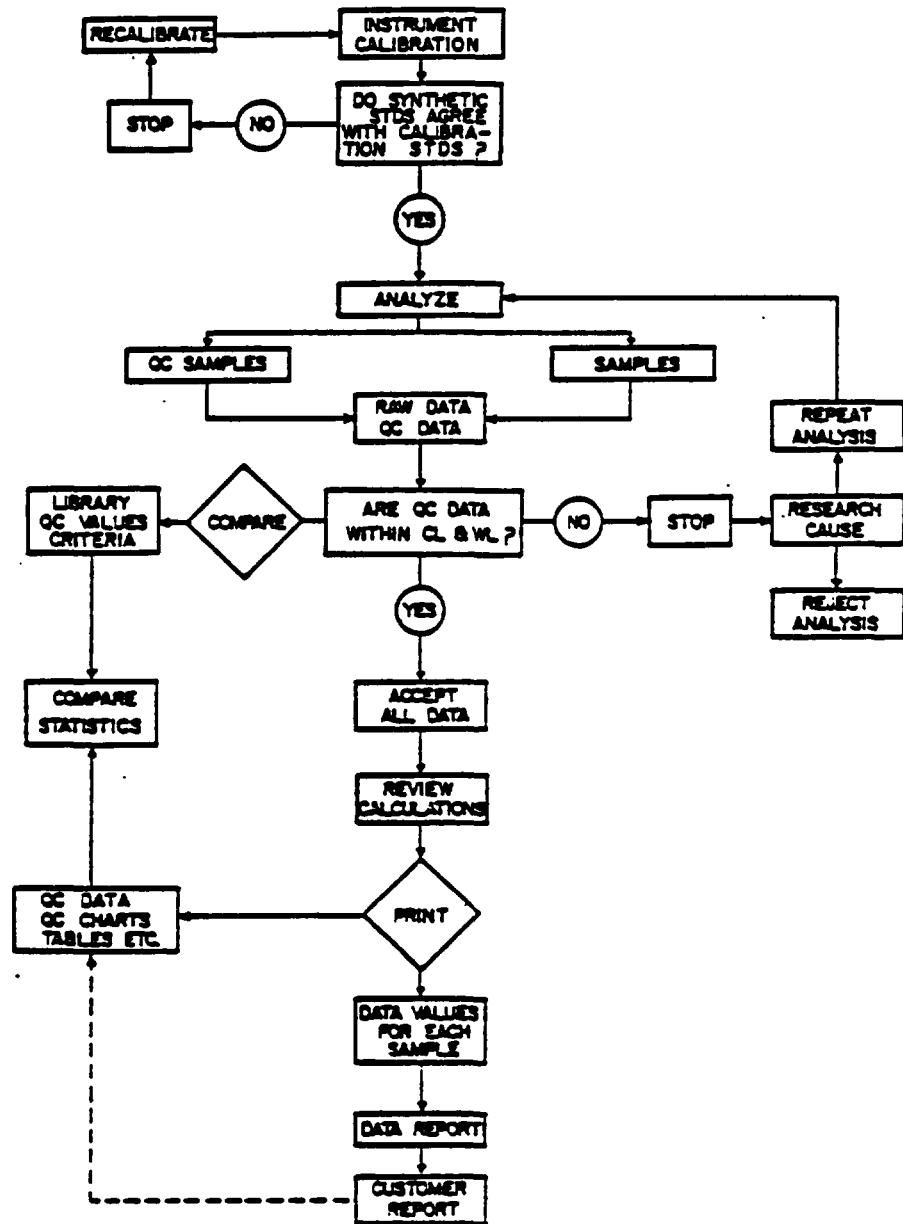
### 3. Other QA/QC Functions

A further ramification of the QA/QC computer management system is the historical evaluations afforded through data storage. Data may be retrieved over long varying time frames providing solid estimates of performance limits for any given analytical parameter. By the same token knowledge of performance limits and the factors that establish them should allow for the improvement of analyses as these factors are identified and removed. Such review is used in the evaluation of new techniques, instruments, and analysts when comparisons are made to the established quality control data bases.

To assist in evaluation and historical review a statistical package is available for measuring the variability of any given data over varying time frames. The Peursonian coefficient of skewness is utilized to quantify variability of percent recoveries, duplicate ratios, and percent of unknown values.

Automatic storage of data, generation of control charts, and data examination through statistics are the tools used to manage the quality control data. The goal of the data management system is a sensitive quality control program which will allow accurate decision making processes and continuous quality of analytical results.

**TABLE XVI**  
**DECISION MAKING PROCESS FOR QA/AC**  
**PROTOCOL AND ANALYSIS OF SAMPLES**



## X. FOR THE CLIENT

The overall importance of our quality control program to the client lies in the fact that we are able to guarantee a certain level of confidence in our analyses. This confidence is expressed through our statistics. As mentioned earlier, we have established our acceptability limits to be plus or minus three times the standard deviation of the mean of the quality control values in each data base. Assuming that the values in the data bases describe a normal distribution, it is known that 99% of the values will fall within the range described by 3 standard deviations of the mean of the distribution. There exists a probability of .99 that any data point will be (plus or minus) 3 times the standard deviation of the mean. This may be described as the 99% confidence interval. We may state, therefore, with 99% certainty, that our quality control data will fall within acceptable limits. As we use quality control data to determine the validity of analyses of client samples, the same confidence interval may be ascribed to such data. The client must be aware, however, that the limits of acceptability are based upon the actual quality control data itself. That data derived from quality control analyses directly reflects the variability of the test. The limits, therefore, will vary as the test varies. Accordingly, the confidence interval of 99% will depict a different range in concentration for each test. The use of the confidence interval provides us with a method of checking the quality of our data and providing the client with some guarantee of validity.

The other facet of our operation which must be described is the ability to adapt our quality control options to the client's specific needs. Quality control parameters, blanks, spiked samples, duplicates,

and the analysis of knowns may increase or decrease in frequency according to the client's wishes. If, for example, there is a concern over contamination, a client may wish to increase the number of blanks from one per ten client samples to two per set. The same applies to spikes, duplicates, and knowns.

If requested, graphs of all quality control data and lists of the statistical information can be made available. The graphs include sample numbers, mean, warning limits and control limits for acceptability (see Figures 6 and 7). The graphs may be formulated to include any desired number of data points for each of the quality control parameters. Statistical lists for data groups include the mean, standard deviation, median, coefficient of skewness and measures of kurtosis. These values can also be modified to comprise varying groups of data points. The variation is related to the time frame the client may wish to relate the data to provide the best description of the validity of analyses on his samples.



## APPENDIX

NLI 001 0483

## KEY FOR DAILY QUALITY CONTROL REPORT

PROJECT NO: denotes client and parameters tested.

SAMPLE: denotes O'Brien & Gere sample ticket number.

MATE: client sample that was spiked or duplicated.

TYPE: Quality control sample type as:

- 1 - blank sample
- 3 - denotes duplicate
- 50 - chemistry spike
- 51 - trace organics spike
- 40 - EPA known concentration

QC VALUE: value obtained for QC sample as blank value; duplicate ratio, percent recoveries for spiked and known samples.

L, WARNING: lower warning limit as  $(-2)$  times the standard deviation of the mean of the last 25 samples.

U, WARNING: upper warning limit as  $(+2)$  times the standard deviation of the mean of the last 25 samples.

SIZE: number of values in data base.

COMMENTS: as written.

**TABLE VII**

**SPIKED RECOVERIES DATA BASE  
FOR GENERATING CONTROL CHARTS & STATISTICS**

HG DATABASE SIZE IS 25 NUMBER OF SAMPLES ARE 54

FIELD SAMPLE VALUE

1	64261	109.000
2	64456	100.000
3	64362	110.000
4	64398	100.000
5	64322	100.000
6	66507	124.000
7	81628	107.000
8	19683	67.000
9	82504	112.000
10	83870	95.000
11	81075	93.000
12	90035	104.440
13	91773	124.000
14	94289	122.000
15	95283	80.000
16	92510	98.000
17	97001	82.000
18	97055	106.000
19	97253	120.000
20	97906	94.000
21	98469	98.000
22	98743	118.000
23	99151	105.000
24	2409	129.000
25	2410	103.000
26	28598	84.711
27	28833	100.000
28	38710	86.364
29	39461	73.333
30	39629	96.000
31	39653	90.625
32	21292	99.160
33	21310	125.000
34	21741	69.841
35	22011	96.061
36	88265	95.522
37	22504	131.657
38	22611	97.222
39	14622	82.759
40	26661	104.687
41	38006	80.202
42	30037	108.333
43	30035	103.448
44	35971	89.756
45	30929	95.455
46	5590	123.529
47	7507	80.851
48	7547	71.897
49	17107	109.091
50	17514	88.462
51	17521	94.667
52	17831	111.735
53	17860	82.453

BENZ

DATABASE SIZE IS 25

NUMBER OF SAMPLES ARE 45

FIELD SAMPLE VALUE

1	29830	109.677
2	23250	87.000
3	44382	99.000
4	5593	97.500
5	43077	100.000
6	5718	120.000
7	5716	100.000
8	9646	94.500
9	9651	103.000
10	9665	92.000
11	17041	104.065
12	17062	89.000
13	17356	95.500
14	17410	83.333
15	17411	90.244
16	17585	105.000
17	17586	105.000
18	17688	105.000
19	17767	100.000
20	17774	102.000
21	17857	77.419
22	17875	87.948
23	17930	99.500
24	18104	102.000
25	18107	105.000
26	18125	88.599
27	18170	105.000
28	18234	80.645
29	18236	84.391
30	18203	93.000
31	18366	105.000
32	18464	105.000
33	18472	105.000
34	50629	91.662
35	50648	100.000
36	50789	91.667
37	50824	108.333
38	50869	100.000
39	50867	100.000
40	50942	100.000
41	50943	105.000
42	51035	105.000
43	51049	100.000
44	51051	105.000
45	51079	91.667

FIELD	SAMPLE	VALUE	FIELD	SAMPLE	VALUE	FIELD	SAMPLE	VALUE	FIELD	SAMPLE	VALUE
1	89582	101.000	50	81561	102.000	103	98317	107.000	158	40838	105.802
2	90104	107.000	51	20670	101.000	104	98850	124.000	159	40837	112.500
3	90559	112.000	52	21982	94.000	105	31724	87.000	160	40836	107.143
4	90736	108.000	53	36918	101.000	106	98950	129.000	161	40835	105.802
5	90824	113.000	54	89124	103.000	107	2412	79.000	162	40834	112.500
6	90893	113.000	55	89075	104.000	108	3383	115.000	163	40833	103.418
7	91032	113.000	56	89182	58.000	109	98880	113.000	164	40832	102.703
8	91033	102.000	57	80915	103.000	110	2409	86.000	165	40831	94.737
9	91125	129.000	58	89443	103.000	111	2412	86.000	166	40830	105.747
10	91222	98.000	59	82401	101.000	112	2409	136.000	167	40829	102.703
11	91430	120.000	60	89100	106.000	113	99339	114.000	168	40828	105.263
12	91448	98.000	61	89692	99.000	114	28104	95.238	169	23519	103.125
13	91457	117.000	62	94693	126.000	115	28277	80.000	170	36773	96.552
14	91503	106.000	63	94674	131.000	116	28369	97.561	171	37917	97.500
15	91542	114.000	64	41561	118.000	117	28487	115.789	172	37969	100.813
16	84864	132.000	65	41567	126.000	118	28633	99.598	173	40283	98.592
17	4301	194.000	66	88024	123.000	119	28847	102.915	174	31498	102.703
18	43127	111.000	67	85080	114.000	120	38623	97.872	175	67826	95.238
19	93046	128.000	68	95155	126.000	121	38664	99.759	176	31033	98.893
20	94518	115.000	69	94963	120.000	122	38885	99.099	177	45425	101.266
21	27732	104.000	70	94976	111.000	123	39186	105.556	178	35804	96.774
22	41728	102.000	71	95312	111.000	124	39268	112.195	179	7591	98.925
23	93079	104.000	72	95365	113.000	125	39520	101.123	180	9686	104.167
24	94428	116.000	73	95466	126.000	126	39870	100.000	181	9687	92.398
25	92360	113.000	74	95500	110.000	127	39959	102.362	182	17061	101.031
26	41899	102.000	75	95579	116.000	128	21162	103.226	183	17396	103.704
27	91596	102.000	76	95778	110.000	129	21515	95.652	184	17544	107.692
28	91650	126.000	77	95820	96.000	130	21641	91.228	185	17569	80.000
29	81738	104.000	78	95871	108.000	131	21857	102.857	186	97676	118.000
30	81810	112.000	79	95956	116.000	132	88134	106.977	187	99663	111.000
31	81913	105.000	80	96229	103.000	133	88288	104.504	188	50601	100.000
32	81992	120.000	81	96230	109.000	134	88399	90.411	189	50606	114.286
33	82095	131.000	82	96231	103.000	135	88464	102.632	190	51103	100.000
34	82235	116.000	83	96497	85.000	136	88600	101.961			
35	19674	110.000	84	96562	108.000	137	88762	107.071			
36	82131	106.000	85	96808	106.000	138	22351	103.226			
37	81425	105.000	86	97014	96.000	139	22429	106.456			
38	82503	114.000	87	97182	124.000	140	22630	106.667			
39	82554	103.000	88	9282	108.000	141	11031	100.000			
40	82573	105.000	89	92617	113.000	142	11211	100.000			
41	83852	110.000	90	97714	99.000	143	40851	86.331			
42	83870	101.000	91	97966	120.000	144	40850	87.640			
43	81003	87.000	92	97907	124.000	145	40849	90.141			
44	84196	103.000	93	98133	121.000	146	40848	92.308			
45	80568	194.000	94	98215	156.000	147	40847	92.453			
46	84381	102.000	95	98243	105.000	148	40846	100.000			
47	84338	107.000	96	98297	109.000	149	40845	98.630			
48	90187	101.000	97	98359	128.000	150	40844	100.000			
49	90364	107.000	98	98496	101.000	151	40843	105.128			
50	90337	100.000	99	98591	79.000	152	40842	100.000			
51	90373	114.000	100	98708	110.000	153	40841	101.923			
52	90364	114.000	101	41562	106.000	154	40840	106.977			
						155	40839	107.143			

**FIGURE 4**

**DAILY QUALITY CONTROL REPORT**

**(SEE KEY)**

PH	N	IPLE	TE	DATE	VALUE	WARNING L.	WARNING	SIZE	COMMENTS
1042-	97-510	18465	0	1	3/15/83				
1042-	97-510	50585	0	1	3/16/83				
1042-	97-510	50714	0	1	3/15/83				
1042-	97-510	50783	0	1	3/15/83				
1042-	97-510	50787	0	1	3/15/83				
1042-	97-510	50790	0	1	3/16/83				
1042-	97-510	50793	0	1	3/16/83				NO SCHEDULED ANALYSES
1042-	97-510	50795	0	1	3/16/83				
1042-	97-510	50820	0	1	3/16/83				NO SCHEDULED ANALYSES
1042-	97-510	50840	0	1	3/16/83				NO SCHEDULED ANALYSES
1042-	97-510	50850	0	1	3/16/83				
1042-	97-510	50851	0	1	3/17/83				
1042-	97-510	50853	0	1	3/17/83				
1042-	97-510	50866	0	1	3/18/83				
1042-	97-510	50868	0	1	3/18/83				
1042-	97-510	50883	0	1	3/21/83				NO SCHEDULED ANALYSES
1042-	97-510	50890	0	1	3/21/83				
1042-	97-510	50891	0	1	3/21/83				
1042-	97-510	50892	0	1	3/21/83				
1042-	97-510	50897	0	1	3/21/83				NO SCHEDULED ANALYSES
1042-	97-510	50899	0	1	3/21/83				
1042-	97-510	50900	0	1	3/21/83				
1042-	97-510	50906	0	1	3/22/83				
	BENZ	<1.			1.000				481
	CHCL3	<1.000			1.000				191
	FREON113	<1.000			1.000				593
	CL3CCH3	<1.000			1.000				608
	CCL4	<1.000			1.000				73
	BRCL2CH	<1.000			1.000				191
	CL3C2H	<1.000			1.000				609
	CLBR2CH	<1.000			1.000				191
	CHBR3	<10.000			10.000				190
	CL4C2	<1.000			1.000				603
	CH2CL2	<1.000			1.000				604
	TOLUENE	<1.000			1.000				482
	M-XYLENE	<1.000			1.000				473
	CLOROBZ	<1.000			1.000				171
	CL4C2H2	<1.000			1.000				66
	DCETAN11	<1.000			1.000				342

NLI 001 0489

DCLEN11	<1.000	1	342
HB	<1.000	1.000	98
LEN12	<1.000	1.000	342
C2H5CL	<1.000	1.000	316
CH2CHCL	<1.000	1.000	315
XYLENES	<1.000	1.000	195
CH3CL	<1.000	1.000	66
CH3BR	<1.000	1.000	66
DCPAN12	<1.000	1.000	66
DCPENT13	<1.000	1.000	66
CL3C2112	<1.000	1.000	66
DCPENC13	<1.000	1.000	66
CLETHR	<10.000	10.000	66

1042- 97-510	50907	0 1 3/22/83		
BENZ	2.		2.000	482
CHCL3	<1.000		1.000	192
FREDN113	<1.000		1.000	594
CL3CCH3	<1.000		1.000	609
CCL4	<1.000		1.000	74
BRCL2CH	<1.000		1.000	192
CL3C2H	<1.000		1.000	610
CLBR2CH	<1.000		1.000	192
CHBR3	<10.000		10.000	191
CL4C2	<1.000		1.000	604
CH2CL2	<1.000		1.000	605
TOLUENE	<1.000		1.000	483
M-XYLENE	<1.000		1.000	474
CLOROHZ	<1.000		1.000	172
CL4C2H2	<1.000		1.000	67
DCETAN11	<1.000		1.000	343
DCETAN12	<1.000		1.000	343
DCLEN11	<1.000		1.000	343
ETHBENZ	<1.000		1.000	99
DCLEN12	<1.000		1.000	343
C2H5CL	<1.000		1.000	317
CH2CHCL	<1.000		1.000	316
XYLENES	<1.000		1.000	196
CH3CL	<1.000		1.000	67
CH3BR	<1.000		1.000	67
DCPAN12	<1.000		1.000	67
DCPENT13	<1.000		1.000	67
CL3C2112	<1.000		1.000	67
DCPENC13	<1.000		1.000	67
CLETHR	<10.000		10.000	67

1042- 97-510 50913 0 1 3/22/83

NO SCHEDULED ANALYSES

1042- 97-510 50700 468 3 3/16/83

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NLI 001 0490

CHCL3	<100.000
FREDN113	<10.000
CCL4	<100.000
BRCL2CH	<100.000
CLBR2CH	<10.000
CHBR3	<100.000
CL4C2	<10.000
CLOROHZ	<1.000
CL4C2H2	<10.000
CH3CL	<10.000
CH3BR	<10.000
DCPAN12	<10.000
DCPENT13	<10.000
CL3C2112	<10.000



**< FLAG - SKIPPED**

NLI 001 0491

CHCL3	<1,000
CHCL3	<1,000
BRCL2CH	<1,000
CLBR2CH	<1,000
CHBR3	<10,000
CL4C2	<1,000
TOLUENE	<1,000
M-XYLENE	<1,000
CLOROBZ	<1,000
CL4C2H2	<1,000
ETHBENZ	<1,000
XYLENES	<1,000
CH3BR	<1,000
DCPAN12	<1,000
DCPENT13	<1,000
CL3C2112	<1,000
DCPENC13	<1,000
CLETHET	<10,000

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1042- 97-510 50854 3058 3 3/18/83

CHCL3	<1,000
CCL4	<1,000
BRCL2CH	<1,000
CLBR2CH	<1,000
CHBR3	<10,000
TOLUENE	<1,000
M-XYLENE	<1,000
CLOROBZ	<1,000
CL4C2H2	<1,000
DCETAN12	<1,000
CH3CL	<10,000
CH3BR	<10,000
DCPAN12	<1,000
DCPENT13	<1,000
CL3C2112	<1,000
DCPENC13	<1,000
CLETHET	<10,000

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1042- 97-510 50856 18315 3 3/16/83

AL <.1

< FLAG - SKIPPED

1042- 97-510 50858 215 3 3/17/83

NO SCHEDULED ANALYSES

1042- 97-510 50860 50660 3 3/17/83

F <.10

< FLAG - SKIPPED

1042- 97-510 50862 18338 3 3/17/83

1042- 97-510 50885 212 3 3/21/83

1042- 97-510 50898 3902 3 3/21/83

BENZ	<100.
CHCL3	<1000,000
FREON113	<1000,000
CCL4	<1000,000
BRCL2CH	<1000,000
CLBR2CH	<1000,000
CHBR3	<10000,000
CL4C2	<1000,000
CH2CL2	<1000,000
TOLUENE	<100,000
M-XYLENE	<100,000
CLOROBZ	<100,000
CL4C2H2	<1000,000

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 < FLAG - SKIPPED

DELTANE	<1000,000
DELTANE	<1000,000
DELTANE	<1000,000
C2H5CL	<1000,000
CH2CHCL	<1000,000
XYLENES	<1000,000
CH3CL	<1000,000
CH3BR	<1000,000
DCPAN12	<1000,000
DCPENT13	<1000,000
CL3C2112	<1000,000
DCPEN13	<1000,000
CLETHEN	<1000,000

1042- 97-510	50800	3	3/21/83
BENZ	<1.		
CHCL3	<1,000		
CCL4	<10,000		
BRCL2CH	<10,000		
CLBR2CH	<1,000		
CHBR3	<10,000		
M-XYLENE	<1,000		
CLOROBZ	<1,000		
CL4C2H2	<1,000		
DCETAN12	<1,000		
ETHBENZ	<1,000		
DCLN12	<1,000		
C2H5CL	<1,000		
CH2CHCL	<1,000		
XYLENES	<1,000		
CH3CL	<1,000		
CH3BR	<1,000		
DCPAN12	<1,000		
DCPENT13	<1,000		
CL3C2112	<1,000		
DCPENC13	<1,000		
CLETHR	<10,000		

[illegible]

CL4C2,F	2,000	2,000	100,000	60,0234	114,7234	37
CH2CL2	2,000					
CH2CL2,F	2,000	2,000	100,000	75,9820	105,6501	31
TOLUENE	2,000	2,000	100,000	77,4026	115,4676	39
TOLUENE,F	2,000					
CLOROHZ	2,000	2,000	100,000	48,9895	128,4501	25
CLOROHZ,F	2,000					
DCETAN11	2,000	2,000	100,000	78,4276	96,3720	27
DCETAN11,F	2,000					
DCETAN12	2,000	2,000	100,000	52,9445	105,6725	33
DCETAN12,F	2,000					
DCLEN11	2,000	2,000	100,000	69,3339	110,3648	33
DCLEN11,F	2,000					
ETHBENZ	2,000	2,000	100,000	79,7258	107,1142	37
ETHBENZ,F	2,000					
DCLEN12	2,000	2,000	100,000	73,1254	102,0690	31
DCLEN12,F	2,000					

1042- 97-510 50869 0 40 3/18/83

1042- 97-510 50645 50605 50 3/16/83

1042- 97-510 50667 17868 50 3/15/83  
TCOLI 0.

QC FILT/UNFILT ANALYSIS NOT DONE

1042- 97-510 50739 50650 50 3/16/83

1042- 97-510 50742 50686 50 3/16/83

1042- 97-510 50792 50604 50 3/16/83

1042- 97-510 50819 50660 50 3/16/83

1042- 97-510 50822 50660 50 3/16/83

1042- 97-510 50826 212 50 3/16/83

1042- 97-510 50828 212 50 3/16/83

1042- 97-510 50855 18173 50 3/16/83

1042- 97-510 50857 214 50 3/17/83

1042- 97-510 50859 18268 50 3/17/83

1042- 97-510 50861 50660 50 3/17/83

1042- 97-510 50863 18338 50 3/17/83

1042- 97-510 50893 213 50 3/21/83  
TC 61.

QC FILT/UNFILT ANALYSIS NOT DONE

11C 52.

QC FILT/UNFILT ANALYSIS NOT DONE

10C 9.

QC FILT/UNFILT ANALYSIS NOT DONE

1042- 97-510 50903 240 50 3/21/83

1042- 97-510 50904 18333 50 3/22/83

NO SCHEDULED ANALYSES

HC 1.0

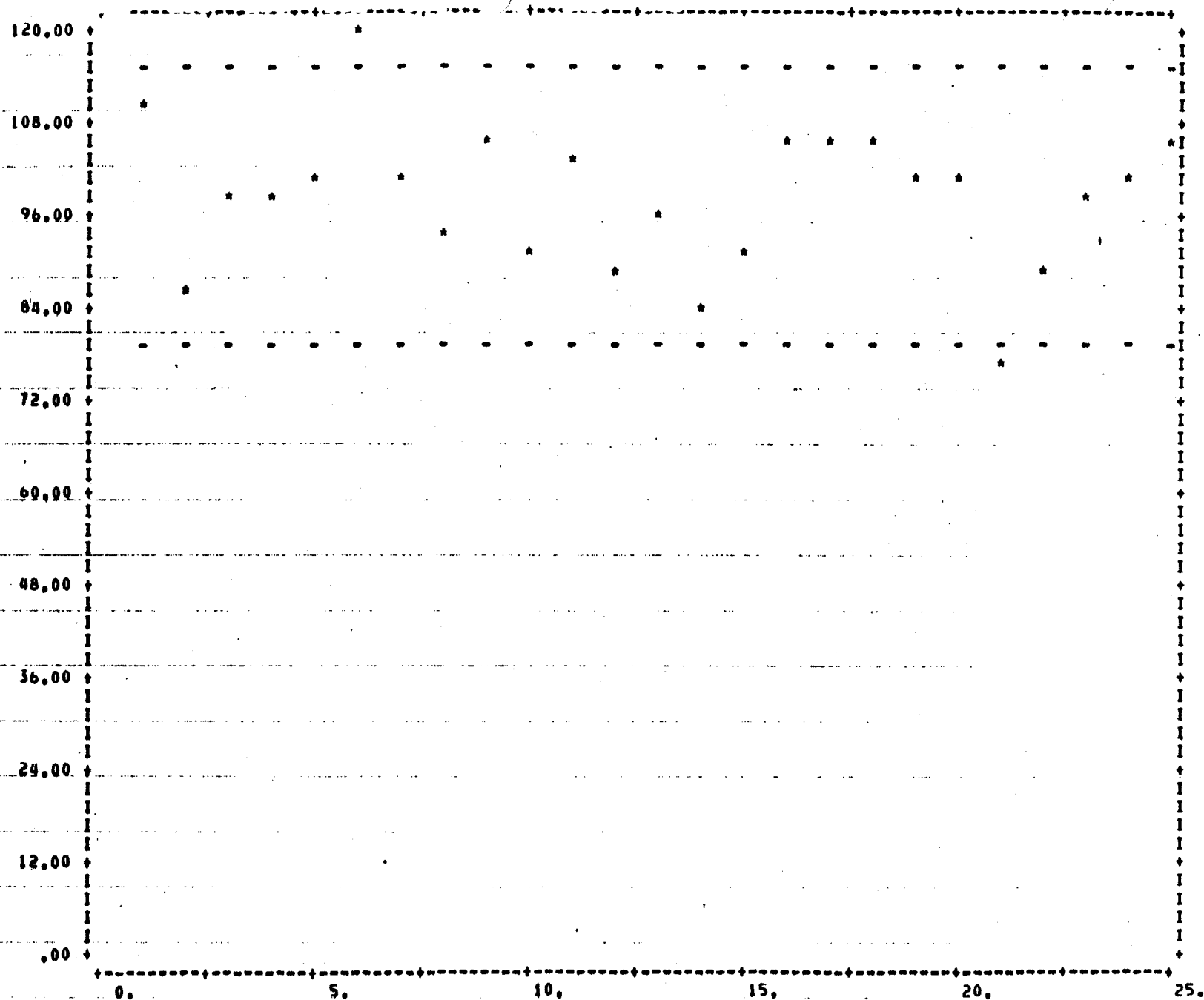
78,947 63,5830 128,5509 54

NLI 001 0494

NO.	2,0								
1042- 97-510	50910	212	50	3/22/83	71.943	71.6096	136.7643	112	
NO2NU3	.52	1.04							
NO2NU3,F	.41								
1042- 97-510	50912	212	50	3/22/83	100.000	79.3854	132.8427	96	
NO2N	.09	<.10							
NO2N,F	.19								
1042- 97-510	50915	210	50	3/22/83	103.333	91.8367	117.0568	156	
ZN	.62	.20							
ZN,F	1.00								
1042- 97-510	50917	267	50	3/22/83	99.259	92.2782	108.3764	149	
CU	.67	.35							
CU,F	1.00								
1042- 97-510	18466	18465	51	3/15/83					
1042- 97-510	50794	50793	51	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50816	50815	51	3/16/83					NO SCHEDULED ANALYSES
1042- 97-510	50884	230	51	3/21/83					NO SCHEDULED ANALYSES
1042- 97-510	50908	50907	51	3/22/83					
BENZ	36.	2.			100.000	70.5872	120.1899	143	
BENZ,F	36.								
TOLUENE	35.000	<1.000			97.222	82.1822	110.5127	148	
TOLUENE,F	36.000								
M-XYLENE	41.000	<1.000			105.128	81.6048	111.9041	135	
M-XYLENE,F	39.000								
CLOROBZ	41.000	<1.000			95.349	57.8437	128.8618	31	
CLOROBZ,F	43.000								
ETHBENZ	41.000	<1.000			100.000	.0000	.0000	7	
ETHBENZ,F	41.000								

FIGURE 5

DAILY QUALITY CONTROL GRAPH  
(SPIKED RECOVERIES)



NLI 001 0497

BENZ

SAMPLES 29830 THROUGH 50867

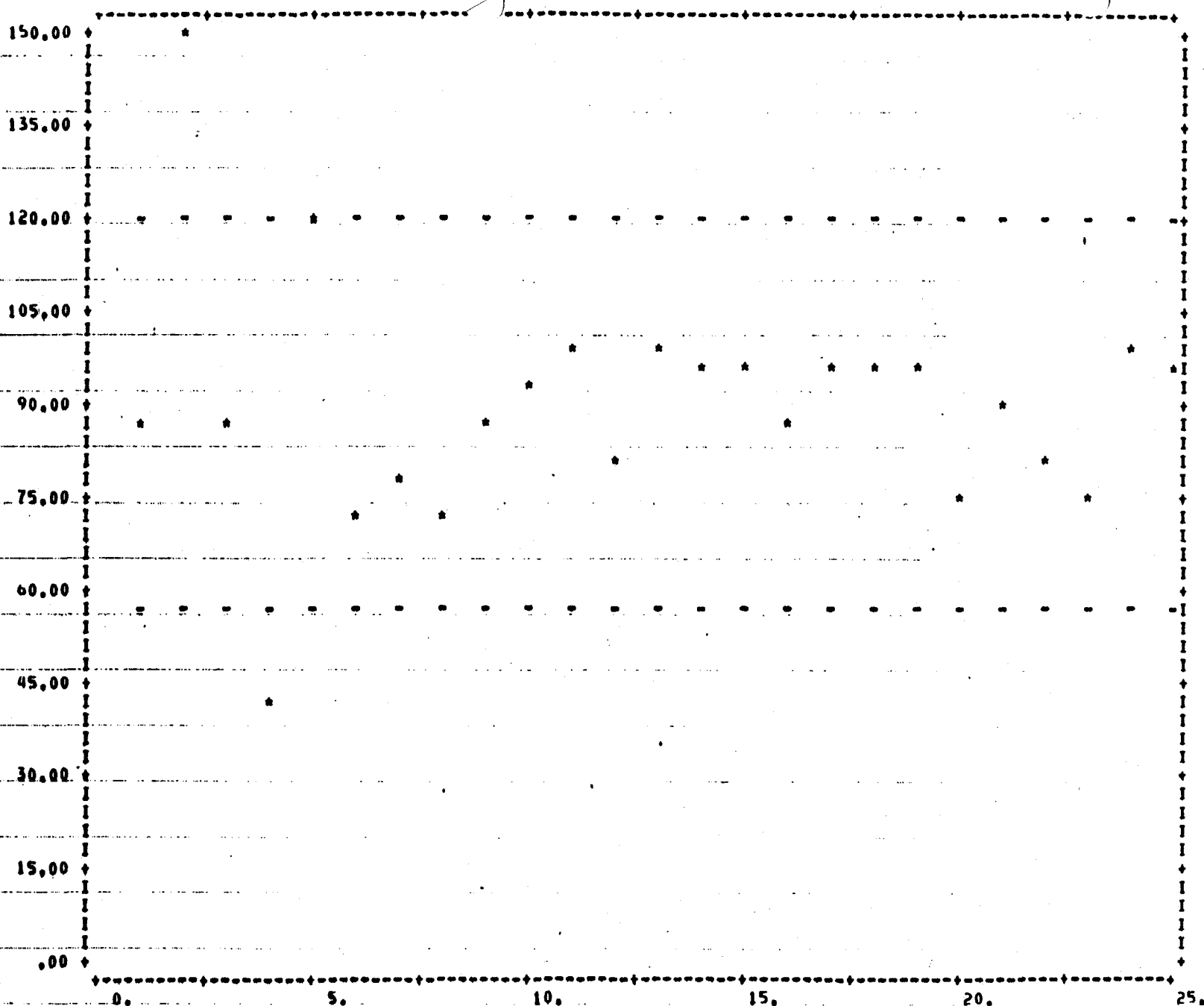
(\*) - QC VALUES (-) -WARNING LIMITS

MEAN = 97.8021 STANDARD DEVIATION = 8.87217

MEDIAN = 100.000 SKEWNESS = -.743181 KURTOSIS = 2.95627

# PERCENT RECOVERY

NLI 001 0498



(\*) - QC VALUES (-) -WARNING LIMITS

MEAN = 88.5001 STANDARD DEVIATION = 15.9869

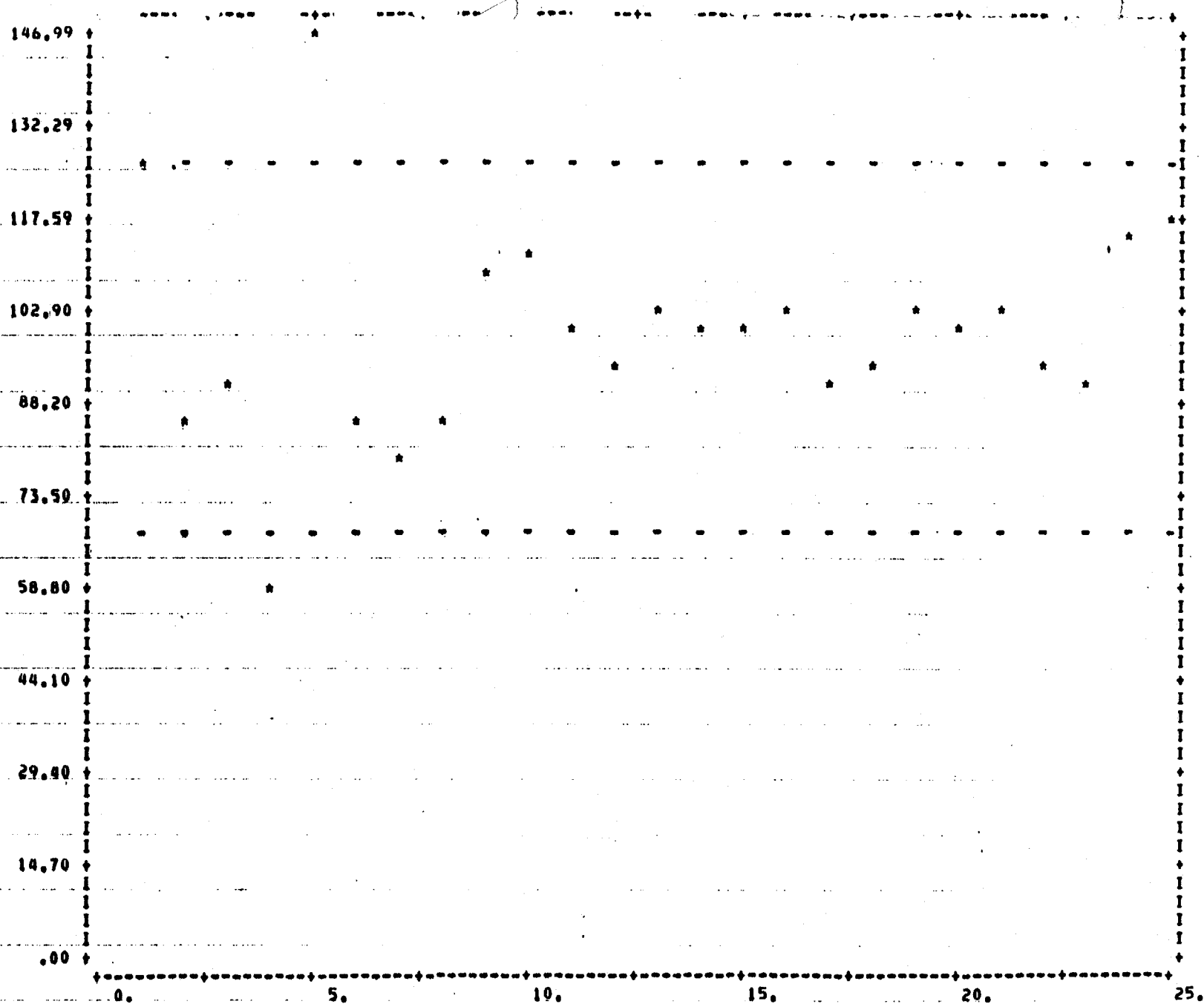
MEDIAN = 87.5000 SKEWNESS = .187665 KURTOSIS = 8.46347

CL3CCH3

SAMPLES 39705 THROUGH 50867



NLI 001 0499



CL3C2H

SAMPLES 39705 THROUGH 50867

(\*) - QC VALUES (-) - WARNING LIMITS  
 MEAN = 97.7033 STANDARD DEVIATION = 14.3505  
 MEDIAN = 100.000 SKEWNESS = -.460136 KURTOSIS = 5.97265

NLI 001 0500

149.78  
134.80  
119.82  
104.85  
89.87  
74.89  
59.91  
44.93  
29.96  
14.98  
.00

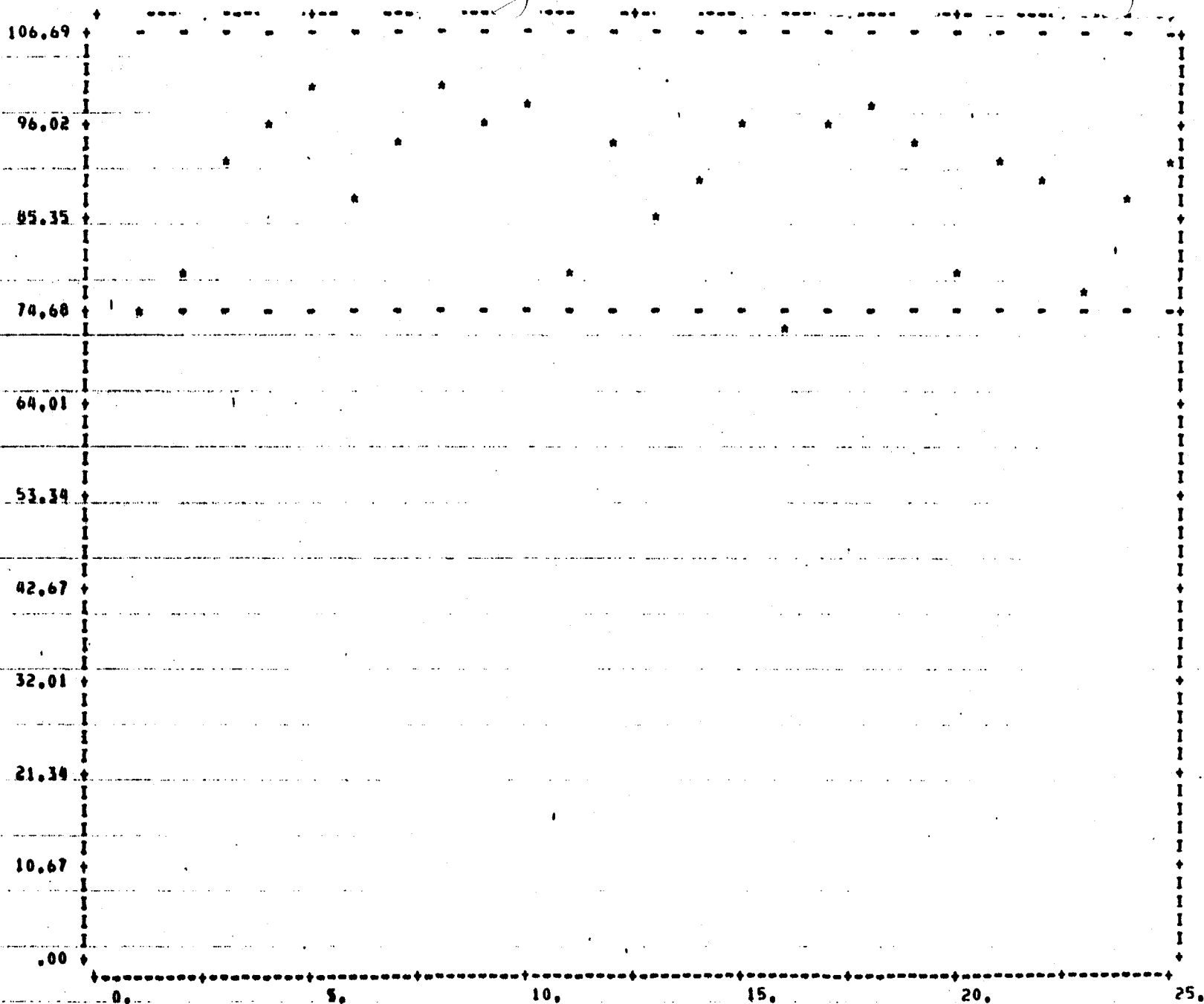
0. 5. 10. 15. 20. 25.

CL4C2

SAMPLES 39705 THROUGH 50867

(\*) = QC VALUES (-) = WARNING LIMITS  
MEAN = 101.354 STANDARD DEVIATION = 19.6983  
MEDIAN = 100.000 SKEWNESS = .206263 KURTOSIS = 4.25532

ILI 001 0501

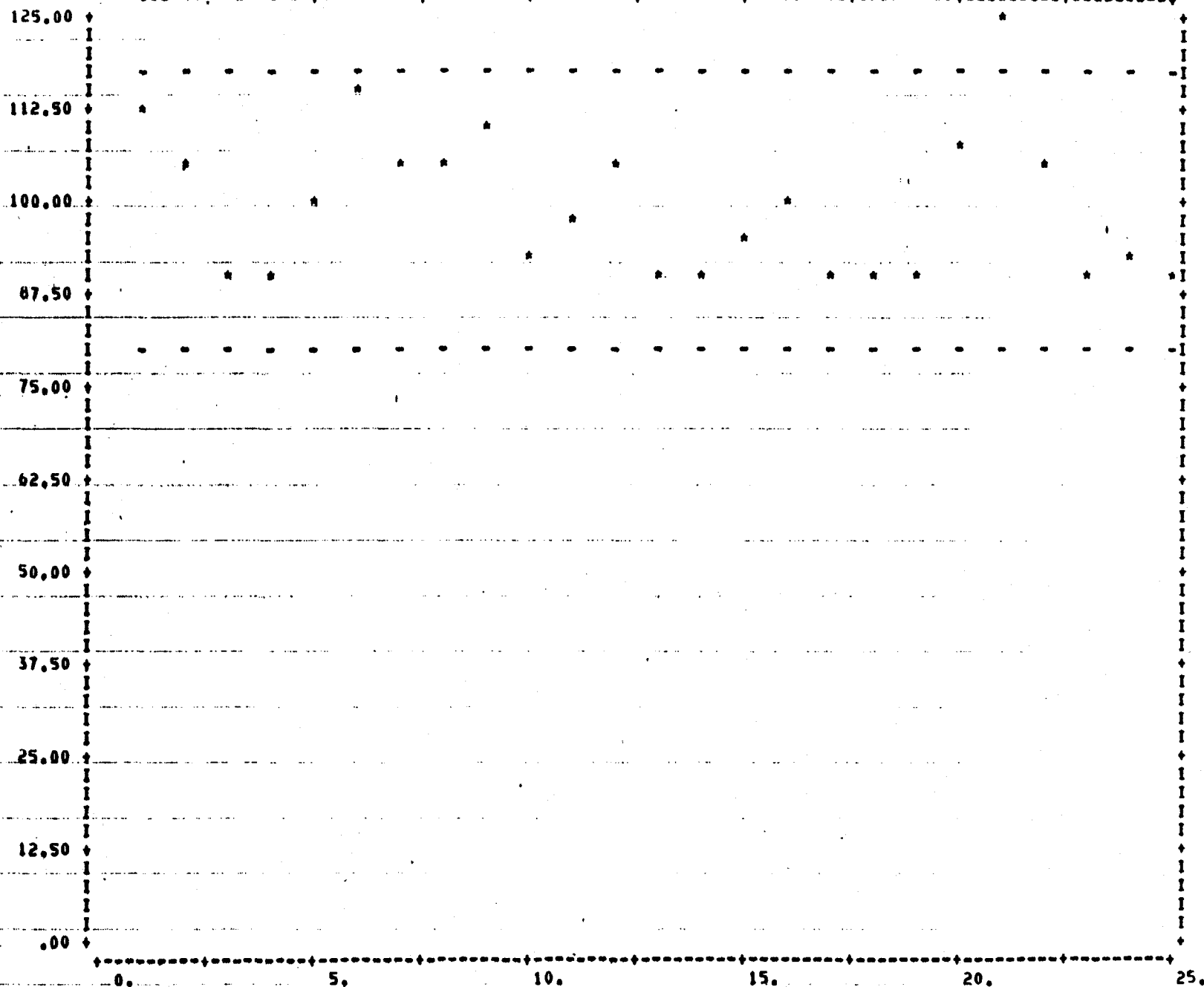


(\*) - QC VALUES (-) - WARNING LIMITS  
 MEAN = 90.7871 STANDARD DEVIATION = 7.94928  
 MEDIAN = 92.5000 SKEWNESS = -.646415 KURTOSIS = 2.47973

CH2CL2

SAMPLES 23250 THROUGH 50867

NLI 001 0502



TOLUENE

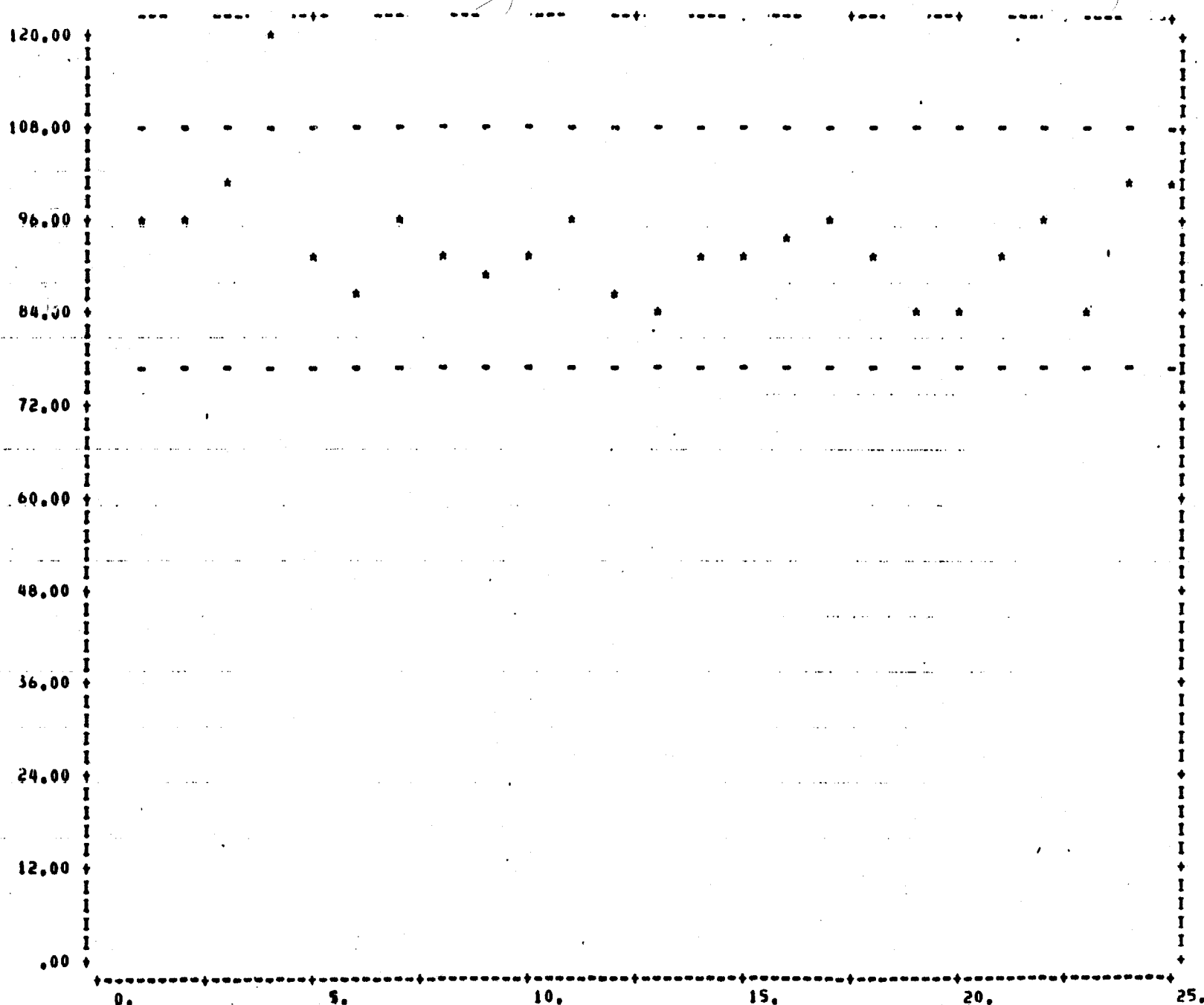
SAMPLES 29830 THROUGH 50867

(\*) - QC VALUES (-) - WARNING LIMITS

MEAN = 98.1067 STANDARD DEVIATION = 9.36984

MEDIAN = 97.0350 SKEWNESS = .103119 KURTOSIS = 3.15778

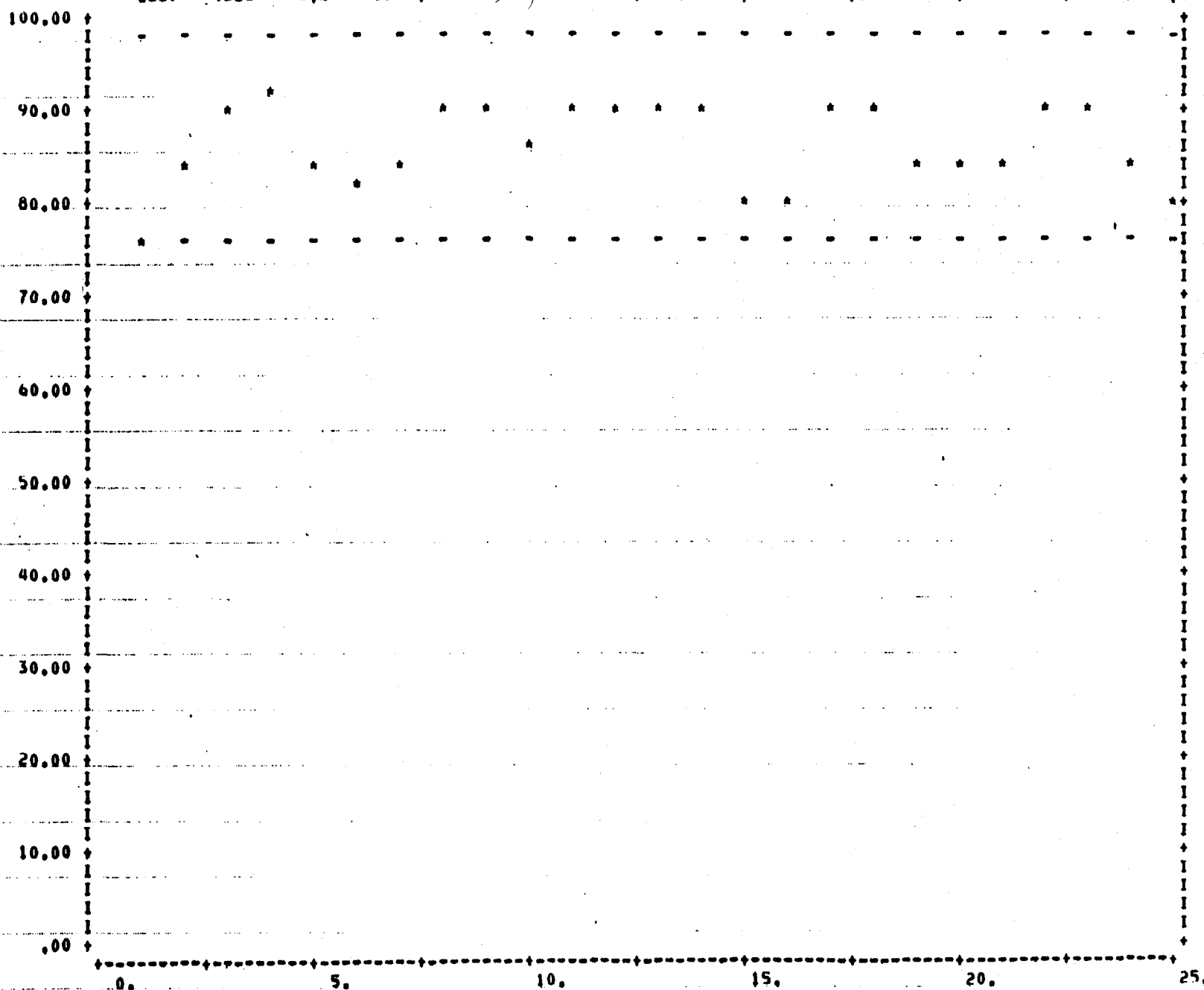
NLI 001 0503



CLOROBZ

SAMPLES 44382 THROUGH 50867

(\*) - QC VALUES (-) - WARNING LIMITS  
 MEAN = 92.7198 STANDARD DEVIATION = 7.43594  
 MEDIAN = 91.0000 SKEWNESS = .693866 KURTOSIS = 8.25299



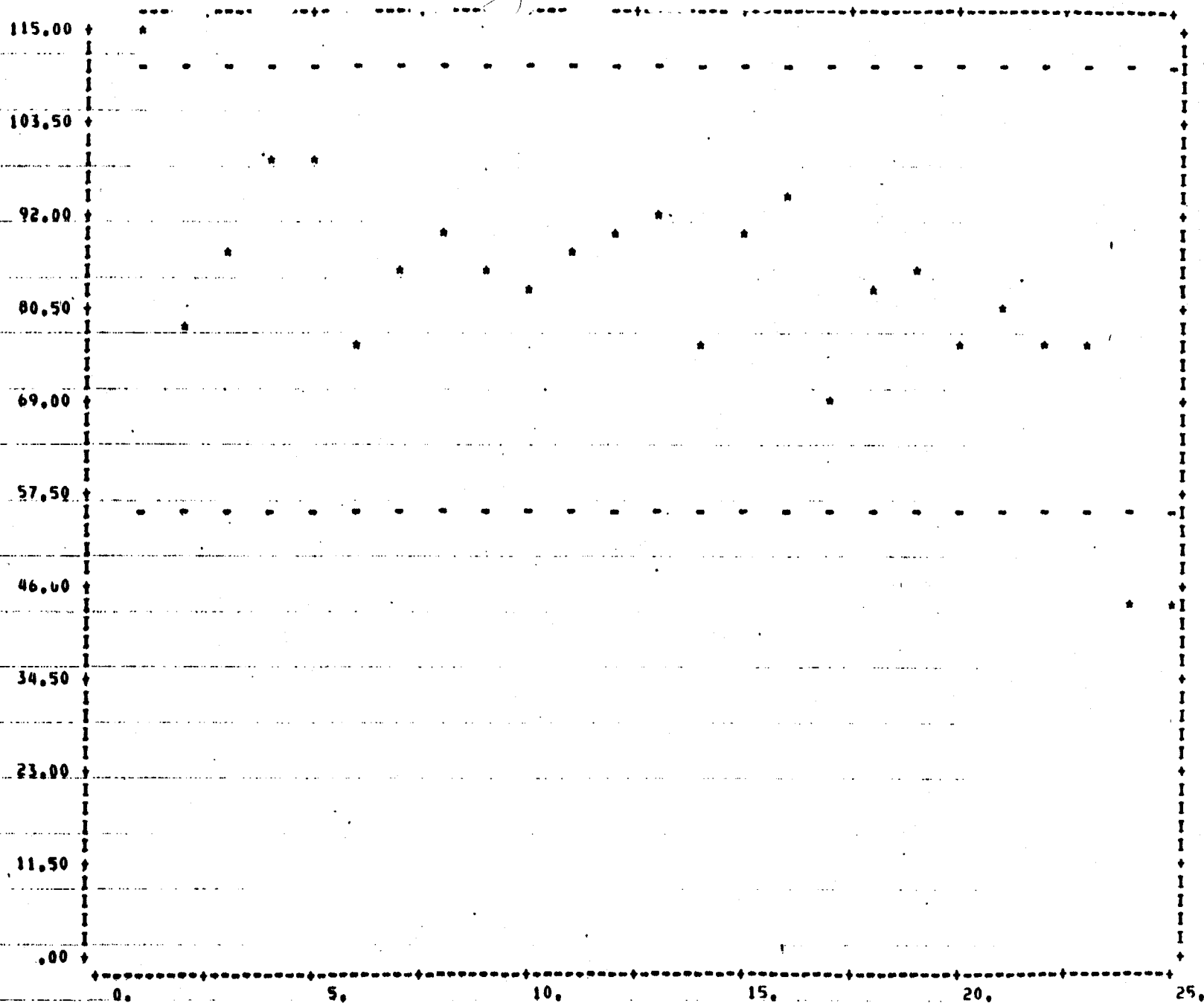
NLI 001 0504

(\*) - QC VALUES (-) -WARNING LIMITS  
 MEAN = 87.4443 STANDARD DEVIATION = 5.44788  
 MEDIAN = 89.0000 SKEWNESS = -.856704 KURTOSIS = 3.53358

DCETAN11 SAMPLES 23250 THROUGH 50867

PERCENT RECOVERY

NLI 001 0505



OCETAN12

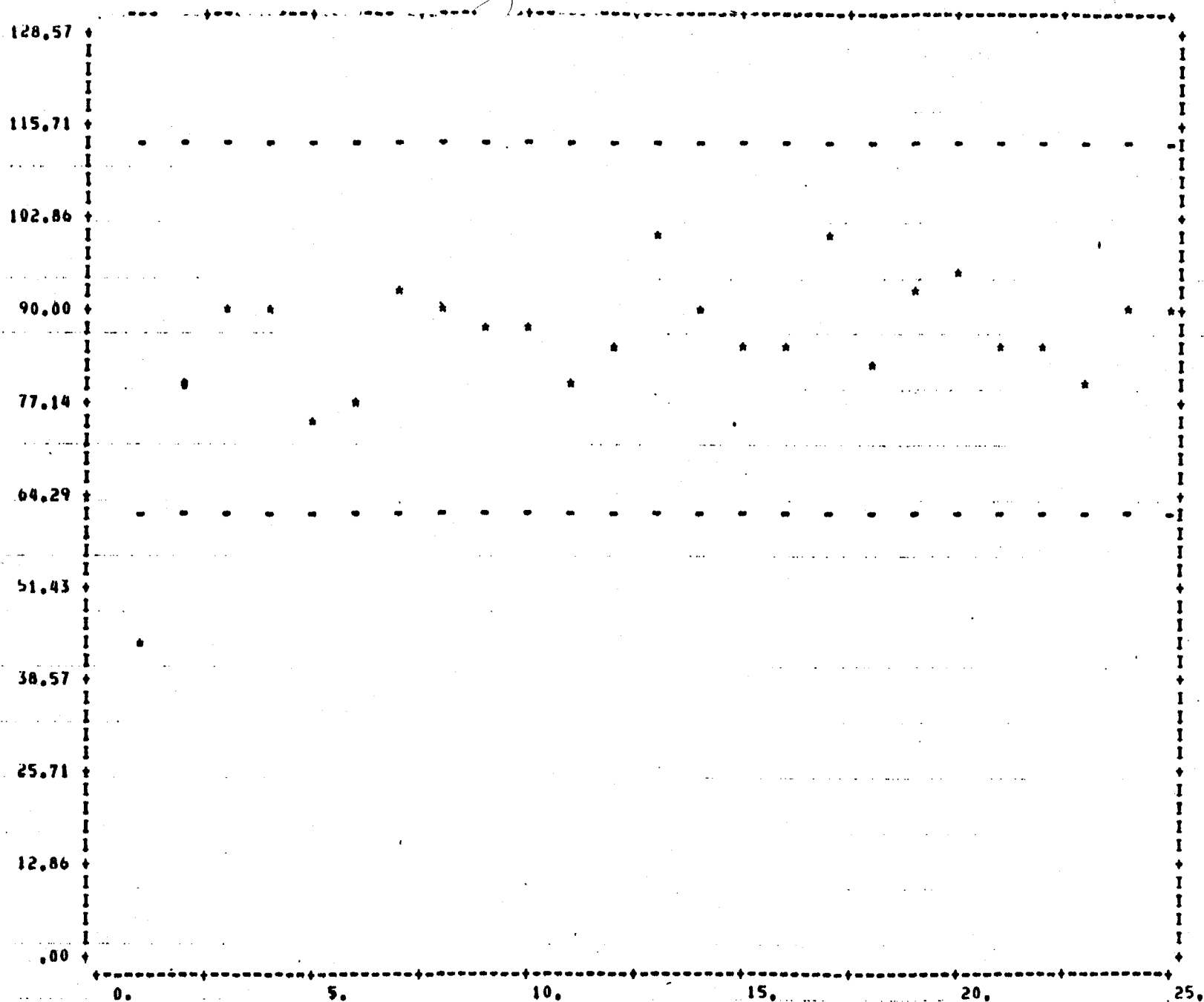
SAMPLES 40299 THROUGH 50867

(\*) - QC VALUES (-) -WARNING LIMITS

MEAN = 82.4609 STANDARD DEVIATION = 14.1964

MEDIAN = 82.5000 SKEWNESS = -.025153E-02 KURTOSIS = 4.72746

NLI 001 0506



DCLEN11

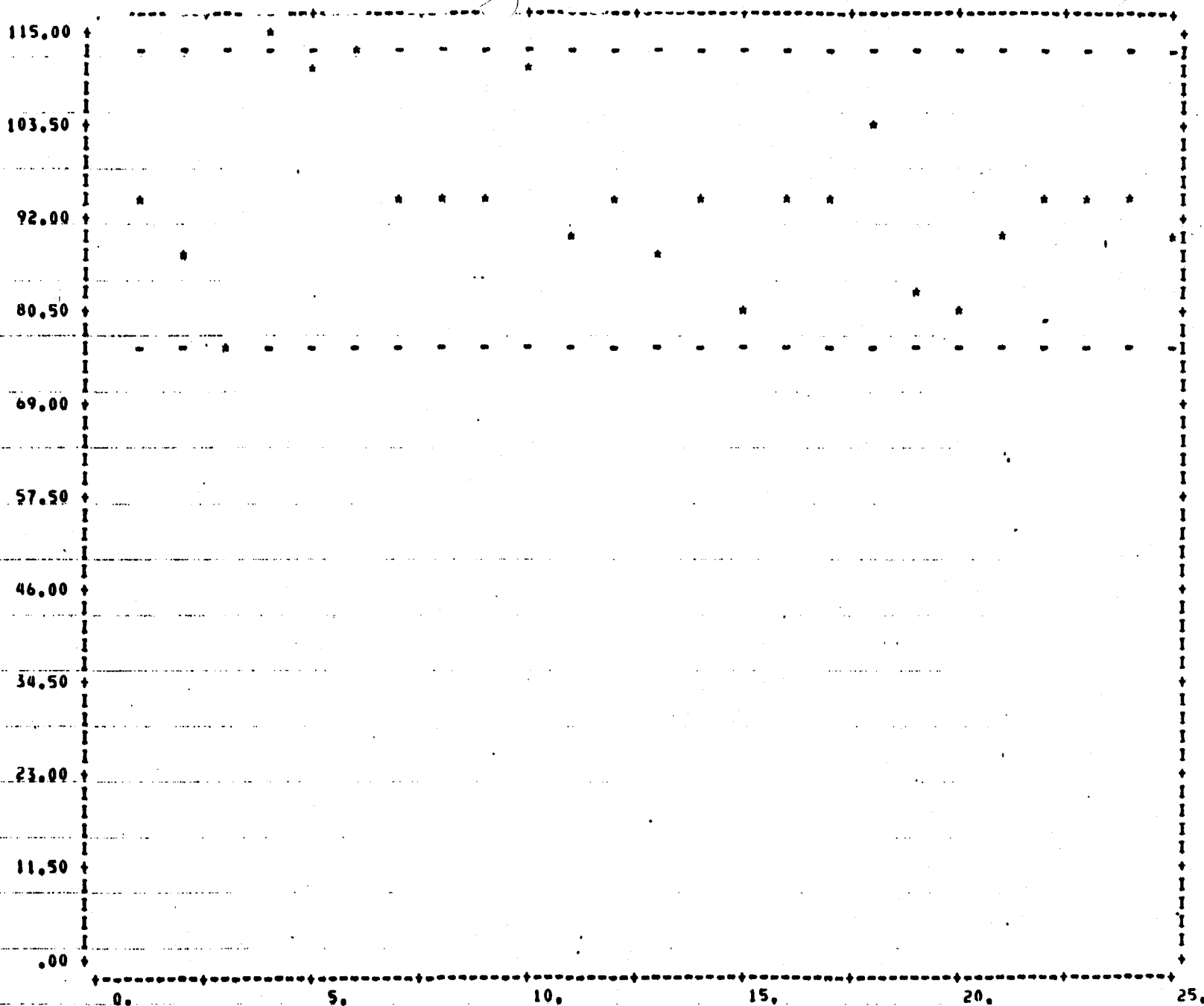
SAMPLES 23250 THROUGH 50867

(\*) - QC VALUES (-) -WARNING LIMITS  
 MEAN = 87.7040 STANDARD DEVIATION = 12.5846  
 MEDIAN = 88.5000 SKEWNESS = -.189742 KURTOSIS = 8.48701



PERCENT RECOVERY

NLI 001 0507



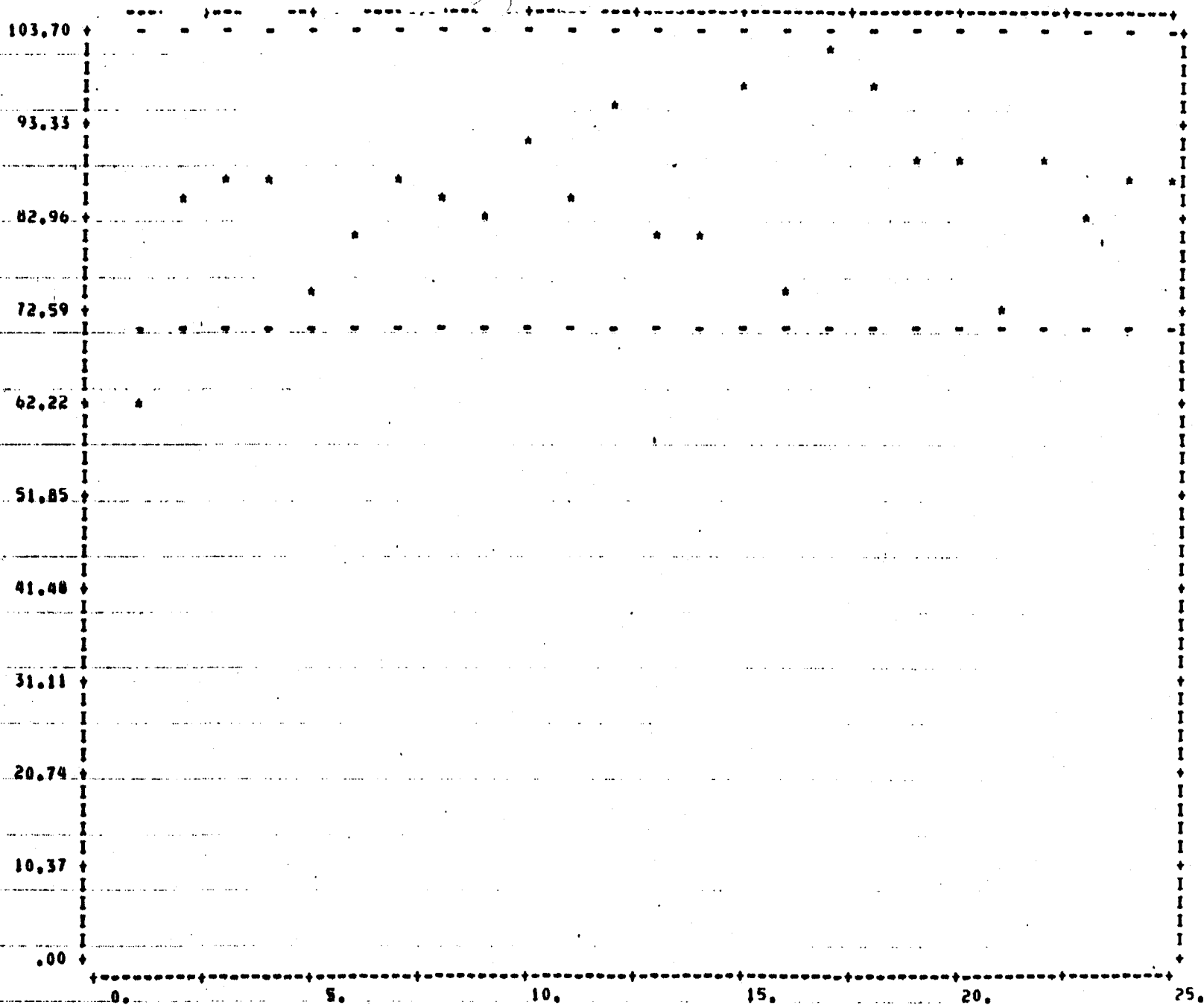
ETHBENZ

SAMPLES 5593 THROUGH 50867

(\*) - QC VALUES (-) -WARNING LIMITS  
 MEAN = 95.0119 STANDARD DEVIATION = 8.99539  
 MEDIAN = 95.0000 SKEWNESS = .196932E-02 KURTOSIS = 3.07061

PERCENT RECOVERY

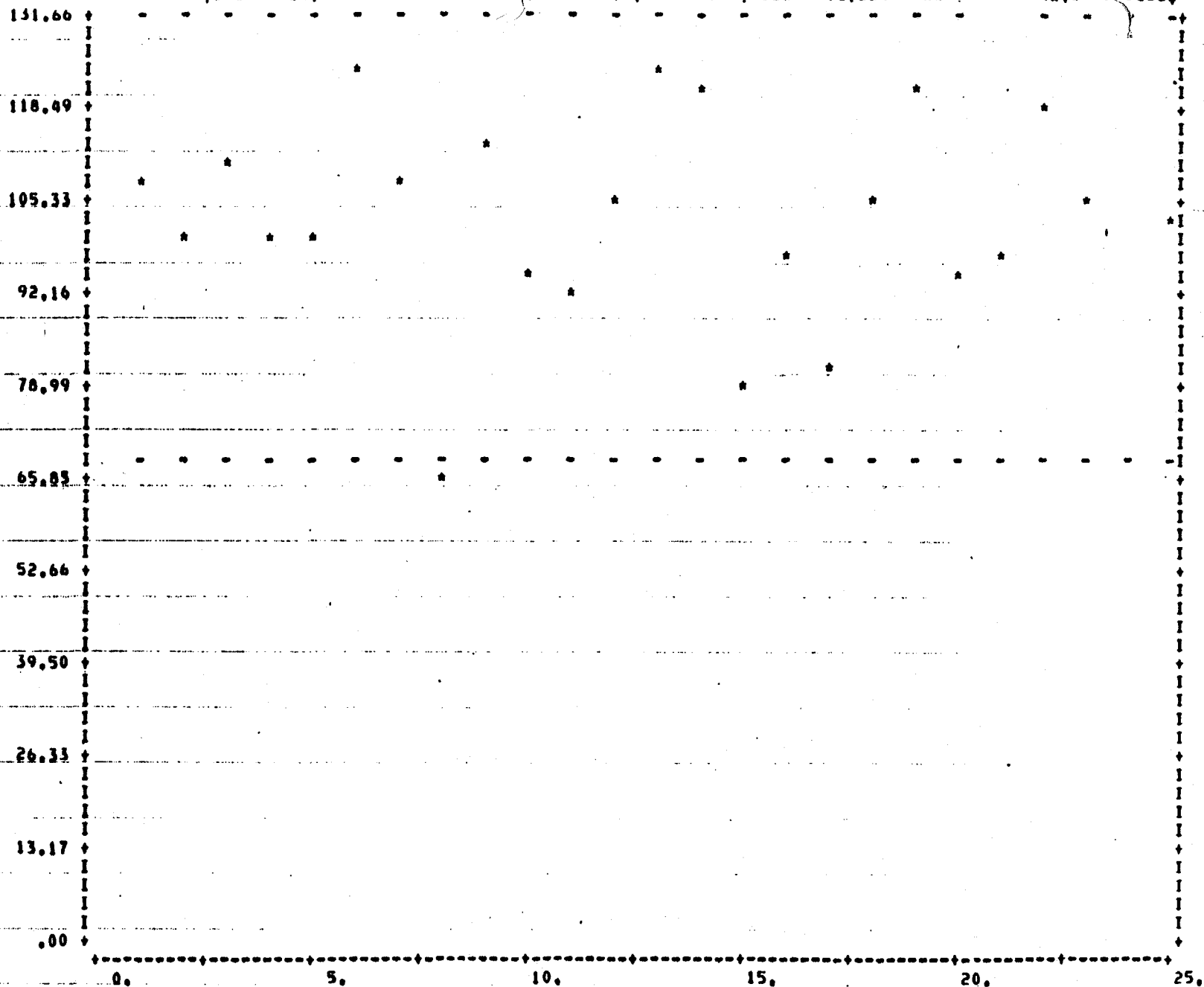
NLI 001 0508



DCLN12 SAMPLES 23250 THROUGH 50867

(\*) = QC VALUES (-) = WARNING LIMITS  
 MEAN = 86.6590 STANDARD DEVIATION = 8.52285  
 MEDIAN = 87.0000 SKEWNESS = -.120015 KURTOSIS = 3.67436

NLI 001 0509



HG

SAMPLES 64261 THROUGH 50904

(\*) = QC VALUES (-) = WARNING LIMITS  
 MEAN = 99.1502 STANDARD DEVIATION = 15.7322  
 MEDIAN = 98.5798 SKEWNESS = .108757 KURTOSIS = 2.46181